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[*N*,*N*′-(1,2-Diphenylethane-1,2-diyl)-bis(pyridine-2-carboxamidato)]nickel(II) diethyl ether hemisolvate

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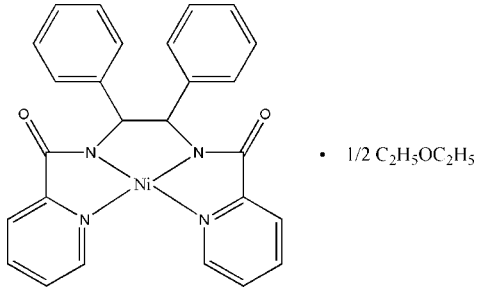
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; disorder in solvent or counterion; R factor = 0.059; wR factor = 0.163; data-to-parameter ratio = 18.7.

In the title compound, $[Ni(C_{26}H_{20}N_4O_2)]\cdot 0.5C_4H_{10}O$, the central metal ion is coordinated by four atoms of the tetradentate picolinamide ligand, forming a slightly distorted square-planar configuration, with an average Ni—N(pyridine) distance of 1.94 Å and an average Ni—N(amide) distance of 1.83 Å. The asymmetric unit contains one half-molecule of diethyl ether; this solvent molecule is disordered across a twofold rotation axis..

Related literature

For related literature, see: Barnes *et al.* (1981); Doukov *et al.* (2002); Fenton *et al.* (1991); Halcrow *et al.* (1994); Mulqi *et al.* (1981); Yang *et al.* (2007).



• 1/2 $C_2H_5OC_2H_5$

Experimental

Crystal data

$[Ni(C_{26}H_{20}N_4O_2)]\cdot 0.5C_4H_{10}O$
M_r = 516.23
Monoclinic, $C2$
a = 21.838 (3) Å
b = 11.1675 (15) Å
c = 11.0443 (15) Å
β = 100.949 (3)°
V = 2644.5 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.77 mm^{-1}
T = 294 (2) K
0.38 × 0.14 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.879, T_{max} = 0.956
12577 measured reflections
6078 independent reflections
3405 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.162
S = 1.01
6078 reflections
325 parameters
17 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}
Absolute structure: Flack (1983), 2840 Friedel pairs
Flack parameter: 0.03 (2)

Table 1
Selected bond lengths (Å).

Ni1—N3	1.8227 (13)	Ni1—N2	1.9410 (17)
Ni1—N1	1.8349 (17)	Ni1—N4	1.9527 (17)

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AV2001).

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[*N*,*N*′-(1,2-Diphenylethane-1,2-diyl)bis(pyridine-2-carboxamidato)]nickel(II) diethyl ether hemisolvate

Li Yang, Dong-Yan Deng, Mei Li and Xiang-Ge Zhou

S1. Comment

Nickel exists in organism as a trace element and it often acts as component or secondary factor of enzyme (Doukov *et al.*, 2002). In the latest two decades, the coordination chemistry of nickel has made a great progress (Halcrow *et al.*, 1994). On the other hand, pyridine carboxamides, being a burgeoning class of multidentate ligands, are available easily from condensation reactions of pyridine carboxylic acid and amine. Continuing of our study in the synthesis and application of complexes containing picolinamide ligands in catalysis (Yang *et al.*, 2007), herein is reported the crystal structure of Ni(II) with chiral pyridine carboxamide ligand, 1,2-bis(2-pyridinecarboxamido)-1,2-diphenylethane(*s,s*-bpdpeH_2).

Selected bond lengths and angles in the title compound are listed in Table 1. Figure 1 shows a perspective drawing of the molecule with atom labeling. In the title compound, there is one solvent ether molecules per two complex molecules. X-Ray crystallography revealed that the nickel ion coordinates with the four nitrogen atoms of picolinamide ligand with Ni–N bond distances ranging from 1.8227 (13) to 1.9527 (17) Å. The sum of the four N—Ni—N angles is 360.48°. The dihedral angle between the two pyridyl rings is less than 2°, the Ni(II) ion seems to form a slightly distorted square plane configuration. Furthermore, the Ni—*N*(amide) distances are shorter than the Ni—*N*(pyridine) distances, which is similar with the reported complex such as [Ni(bcph)](H_2bpch=1, 2-bis(2-pyridinecarboxamido)-1, 2- cyclohexane) with Ni—*N*(amide)=1.87 Å and Ni—*N*(pyridine)=1.94 Å (Mulqi *et al.*, 1981).

S2. Experimental

The ligand was prepared by a previously described method (Fenton *et al.*, 1991). The title complex was obtained analogous to (Barnes *et al.*, 1981). Single crystals suitable for X-ray analysis were obtained by slow diffusion of diethyl ether into a DMF solution of the complex. Selected IR data (KBr, cm^{-1}): 2980 (*m*), 1650 (amide I band, s), 1610(amide II band, s), 1470 (*s*), 1360 (*s*), 1120(*s*), 1020 (*m*), 870 (*m*). Analysis calculated for $C_{26}H_{20}N_4NiO_2$: C 65.17, H 4.21, N 11.69%; found: C 65.23, H 4.43, N 11.54%. MS (FAB): 479([Ni(bpdpe)]$^+$).

S3. Refinement

All H atoms of the complex were positioned geometrically and refined as riding, with C—H = 0.93 Å(aromatic) and 0.98 Å(methylene) with U_{iso}(H) =1.2Ueq(aromatic, methylene).

When solvent disorder was treated, there are 4 larger electron density peaks in different electron density map (Fourier synthesis). And two peaks are located on special positions. These special positions are considered as disorder oxygen atom of ether, O3 and O3′. When these peaks are grown, the whole ether molecule was got. O3 and O3′ have occupancy factor as 0.5 defined by special position.

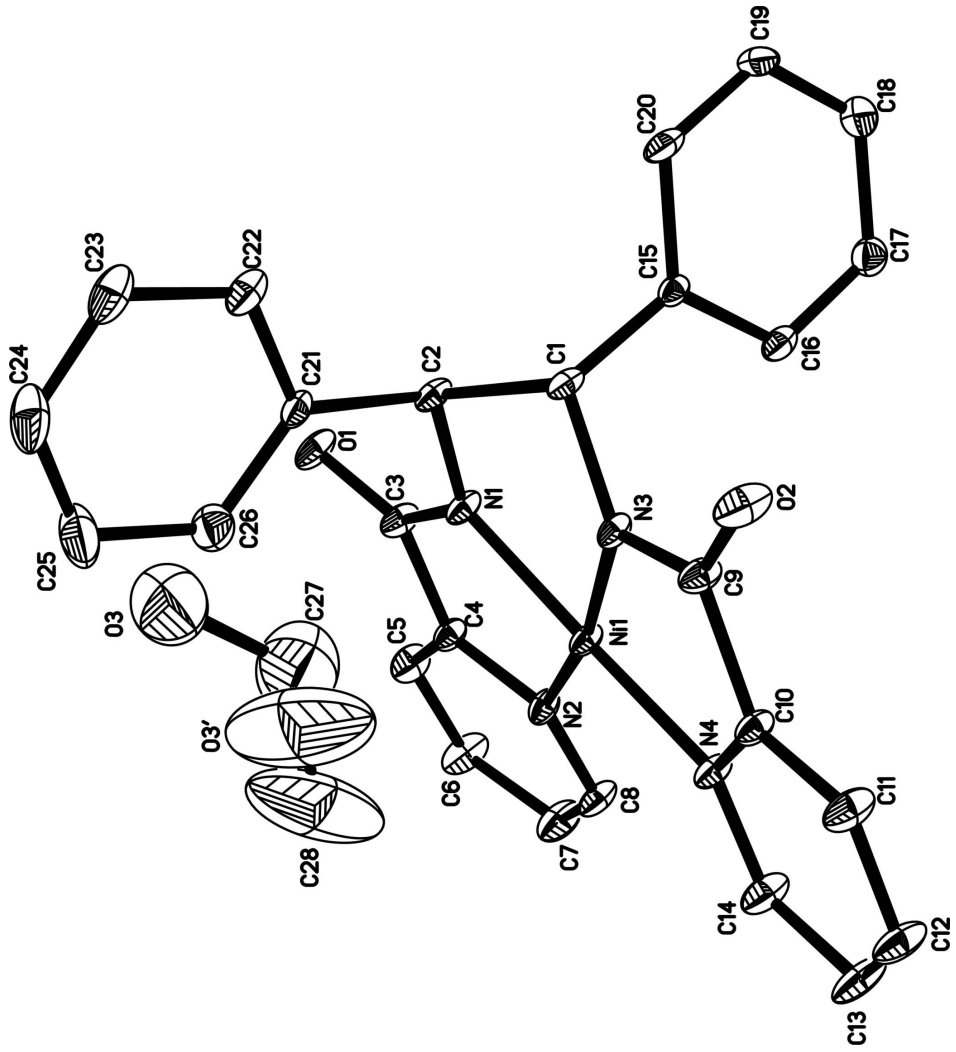

Figure 1

1 A view of $[Ni(C_{26}H_{20}N_4O_2)].1/2(C_2H_5OC_2H_5)$, with displacement ellipsoids drawn at the 30% propability level.

[*N*,*N*′-(1,2-Diphenylethane-1,2-diyl)bis(pyridine-2-carboxamidato)]nickel(II) diethyl ether hemisolvate

Crystal data

$[Ni(C_{26}H_{20}N_4O_2)]\cdot 0.5C_4H_{10}O$
$M_r = 516.23$
Monoclinic, *C*2
$a = 21.838\ (3)$ Å
$b = 11.1675\ (15)$ Å
$c = 11.0443\ (15)$ Å
$\beta = 100.949\ (3)°$
$V = 2644.5\ (6)$ Å^3
$Z = 4$
$F(000) = 1076$
$D_x = 1.297$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4983 reflections
$\theta = 1–27.5°$
$\mu = 0.77$ mm^{-1}
$T = 294$ K
Needle, green
0.38 × 0.14 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.879, T_{max} = 0.956
12577 measured reflections
6078 independent reflections
3405 reflections with $I > 2\sigma(I)$
R_{int} = 0.067
θ_{max} = 27.6°, θ_{min} = 2.7°
h = −28→28
k = −14→14
l = −14→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.162
S = 1.01
6078 reflections
325 parameters
17 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
$w = 1/[\sigma^2(F_o^2) + (0.067P)^2]$ and $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.007
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}
Absolute structure: Flack (1983)
Absolute structure parameter: 0.03 (2)

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	0.685445 (11)	0.41062 (2)	0.575362 (19)	0.03992 (6)	
O1	0.68109 (9)	0.75302 (12)	0.48680 (13)	0.0649 (6)	
O2	0.71735 (10)	0.29814 (15)	0.91975 (14)	0.0807 (7)	
N1	0.69128 (8)	0.57385 (15)	0.59271 (15)	0.0448 (6)	
N2	0.67548 (8)	0.44546 (14)	0.40053 (15)	0.0463 (6)	
N3	0.70417 (7)	0.41280 (19)	0.74336 (12)	0.0404 (4)	
N4	0.67459 (9)	0.23904 (15)	0.59807 (15)	0.0472 (6)	
C1	0.73171 (10)	0.52214 (17)	0.80298 (19)	0.0427 (6)	
H1	0.7199	0.5305	0.8839	0.051*	
C2	0.70105 (11)	0.62494 (17)	0.71610 (18)	0.0437 (7)	
H2	0.7313	0.6904	0.7202	0.052*	
C3	0.68186 (11)	0.64212 (19)	0.49332 (19)	0.0501 (7)	
C4	0.67389 (11)	0.56614 (19)	0.37879 (19)	0.0455 (7)	
C5	0.66656 (13)	0.6111 (2)	0.2626 (2)	0.0642 (9)	
H5	0.6622	0.6933	0.2506	0.077*	

C6	0.66545 (15)	0.5382 (2)	0.1635 (2)	0.0763 (10)	
H6	0.6618	0.5691	0.0843	0.092*	
C7	0.66992 (12)	0.4171 (3)	0.18509 (18)	0.0763 (8)	
H7	0.6700	0.3642	0.1201	0.092*	
C8	0.67431 (12)	0.37406 (18)	0.30362 (19)	0.0579 (8)	
H8	0.6765	0.2917	0.3163	0.069*	
C9	0.70290 (12)	0.3139 (2)	0.8047 (2)	0.0528 (8)	
C10	0.68154 (12)	0.21015 (19)	0.7192 (2)	0.0480 (7)	
C11	0.67415 (14)	0.0961 (2)	0.7604 (2)	0.0704 (9)	
H11	0.6793	0.0798	0.8443	0.084*	
C12	0.65897 (14)	0.0069 (2)	0.6752 (2)	0.0777 (10)	
H12	0.6520	−0.0704	0.7008	0.093*	
C13	0.65399 (16)	0.0315 (2)	0.5531 (3)	0.0847 (11)	
H13	0.6464	−0.0294	0.4947	0.102*	
C14	0.66052 (14)	0.1488 (2)	0.5177 (2)	0.0718 (10)	
H14	0.6549	0.1660	0.4339	0.086*	
C15	0.80143 (10)	0.52376 (17)	0.81812 (17)	0.0390 (6)	
C16	0.83346 (10)	0.45226 (18)	0.74753 (19)	0.0507 (7)	
H16	0.8115	0.4001	0.6894	0.061*	
C17	0.89797 (11)	0.4584 (2)	0.7635 (2)	0.0614 (8)	
H17	0.9189	0.4098	0.7163	0.074*	
C18	0.93127 (12)	0.5359 (2)	0.8487 (2)	0.0700 (10)	
H18	0.9745	0.5400	0.8592	0.084*	
C19	0.90017 (11)	0.6058 (2)	0.9169 (2)	0.0680 (9)	
H19	0.9224	0.6580	0.9747	0.082*	
C20	0.83604 (11)	0.60110 (19)	0.9019 (2)	0.0579 (8)	
H20	0.8157	0.6509	0.9492	0.070*	
C21	0.64296 (11)	0.67357 (18)	0.7536 (2)	0.0484 (7)	
C22	0.64900 (12)	0.7353 (2)	0.86491 (19)	0.0651 (9)	
H22	0.6886	0.7457	0.9124	0.078*	
C23	0.59732 (12)	0.7826 (3)	0.9082 (2)	0.0837 (10)	
H23	0.6023	0.8222	0.9835	0.100*	
C24	0.54060 (14)	0.7686 (3)	0.8374 (2)	0.1052 (12)	
H24	0.5060	0.7988	0.8649	0.126*	
C25	0.53215 (14)	0.7107 (3)	0.7252 (3)	0.1189 (15)	
H25	0.4926	0.7042	0.6766	0.143*	
C26	0.58374 (12)	0.6620 (3)	0.6855 (3)	0.0820 (11)	
H26	0.5779	0.6207	0.6110	0.098*	
C28	0.5081 (3)	0.3730 (6)	0.7884 (4)	0.364 (3)	
H28A	0.5451	0.3351	0.7625	0.545*	
H28B	0.4932	0.4332	0.7310	0.545*	
H28C	0.4779	0.3102	0.7870	0.545*	
C27	0.5289 (3)	0.4225 (3)	0.9168 (4)	0.251 (3)	
H27A	0.5430	0.3517	0.9655	0.301*	
H27B	0.5654	0.4671	0.9113	0.301*	
O3	0.5000	0.4919 (6)	1.0000	0.219 (6)	0.50
O3′	0.5000	0.3518 (7)	1.0000	0.380 (6)	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.06559 (13)	0.02383 (9)	0.03095 (10)	0.00039 (17)	0.01070 (9)	0.00210 (15)
O1	0.1240 (13)	0.0250 (8)	0.0457 (9)	0.0052 (9)	0.0160 (9)	0.0088 (7)
O2	0.1566 (16)	0.0453 (9)	0.0339 (8)	−0.0197 (11)	0.0019 (10)	0.0124 (8)
N1	0.0760 (12)	0.0243 (8)	0.0353 (9)	0.0007 (9)	0.0135 (9)	0.0004 (7)
N2	0.0710 (11)	0.0316 (10)	0.0374 (9)	0.0087 (8)	0.0134 (8)	0.0049 (7)
N3	0.0659 (9)	0.0242 (6)	0.0322 (7)	0.0023 (13)	0.0121 (6)	0.0067 (11)
N4	0.0769 (13)	0.0262 (9)	0.0393 (9)	−0.0040 (9)	0.0135 (9)	−0.0007 (8)
C1	0.0740 (14)	0.0244 (9)	0.0277 (10)	−0.0028 (10)	0.0046 (10)	−0.0016 (8)
C2	0.0765 (14)	0.0247 (10)	0.0303 (10)	−0.0015 (10)	0.0116 (10)	−0.0004 (9)
C3	0.0876 (16)	0.0279 (10)	0.0345 (11)	−0.0060 (11)	0.0106 (11)	0.0093 (9)
C4	0.0681 (14)	0.0292 (10)	0.0391 (11)	0.0008 (10)	0.0100 (11)	0.0042 (9)
C5	0.1020 (19)	0.0462 (14)	0.0438 (13)	0.0003 (14)	0.0126 (13)	0.0142 (11)
C6	0.155 (2)	0.0498 (14)	0.0255 (11)	−0.0059 (16)	0.0196 (14)	0.0090 (11)
C7	0.1395 (19)	0.0557 (13)	0.0351 (10)	0.003 (2)	0.0202 (12)	−0.0076 (17)
C8	0.1057 (18)	0.0313 (12)	0.0371 (11)	0.0017 (11)	0.0143 (12)	−0.0059 (9)
C9	0.0805 (16)	0.0366 (11)	0.0392 (12)	−0.0092 (12)	0.0064 (12)	0.0019 (11)
C10	0.0764 (15)	0.0277 (10)	0.0385 (11)	−0.0036 (11)	0.0071 (11)	0.0032 (10)
C11	0.126 (2)	0.0362 (12)	0.0477 (14)	−0.0156 (14)	0.0135 (15)	0.0124 (11)
C12	0.138 (2)	0.0313 (12)	0.0627 (16)	−0.0212 (15)	0.0159 (16)	0.0045 (12)
C13	0.164 (3)	0.0283 (12)	0.0686 (17)	−0.0175 (16)	0.0389 (17)	−0.0009 (12)
C14	0.130 (2)	0.0402 (13)	0.0430 (14)	−0.0073 (16)	0.0109 (15)	−0.0037 (12)
C15	0.0572 (13)	0.0281 (10)	0.0299 (10)	0.0041 (10)	0.0038 (10)	0.0008 (9)
C16	0.0786 (15)	0.0347 (11)	0.0374 (11)	0.0045 (11)	0.0078 (11)	−0.0029 (9)
C17	0.0718 (16)	0.0565 (15)	0.0536 (14)	0.0109 (12)	0.0061 (13)	−0.0005 (12)
C18	0.0674 (16)	0.0630 (16)	0.0749 (18)	0.0069 (15)	0.0016 (15)	0.0033 (15)
C19	0.0727 (17)	0.0471 (14)	0.0736 (18)	−0.0122 (13)	−0.0130 (15)	−0.0155 (13)
C20	0.0940 (18)	0.0326 (11)	0.0450 (12)	−0.0006 (13)	0.0075 (13)	−0.0119 (10)
C21	0.0674 (14)	0.0300 (11)	0.0467 (12)	0.0131 (11)	0.0082 (11)	−0.0017 (10)
C22	0.1067 (19)	0.0447 (14)	0.0461 (13)	0.0150 (14)	0.0198 (13)	−0.0005 (12)
C23	0.134 (2)	0.0629 (16)	0.0640 (15)	0.0292 (17)	0.0439 (14)	0.0049 (14)
C24	0.111 (2)	0.134 (3)	0.0792 (18)	0.0655 (19)	0.0387 (15)	0.0075 (19)
C25	0.081 (2)	0.149 (3)	0.126 (3)	0.053 (2)	0.017 (2)	−0.004 (3)
C26	0.0860 (19)	0.091 (2)	0.0678 (18)	0.0157 (17)	0.0106 (16)	−0.0138 (16)
C28	0.301 (5)	0.388 (7)	0.344 (7)	−0.243 (5)	−0.087 (6)	0.170 (6)
C27	0.293 (6)	0.249 (7)	0.188 (5)	0.067 (7)	−0.011 (5)	−0.015 (6)
O3	0.167 (8)	0.173 (9)	0.306 (14)	0.000	0.022 (9)	0.000
O3′	0.382 (16)	0.400 (3)	0.398 (3)	−0.26 (2)	0.176 (13)	−0.13 (4)

Geometric parameters (Å, º)

Ni1—N3	1.8227 (13)	C13—C14	1.383 (4)
Ni1—N1	1.8349 (17)	C13—H13	0.9300
Ni1—N2	1.9410 (17)	C14—H14	0.9300
Ni1—N4	1.9527 (17)	C15—C20	1.381 (3)
O1—C3	1.241 (3)	C15—C16	1.394 (3)

O2—C9	1.263 (3)	C16—C17	1.388 (3)
N1—C3	1.320 (3)	C16—H16	0.9300
N1—C2	1.455 (3)	C17—C18	1.380 (3)
N2—C8	1.331 (3)	C17—H17	0.9300
N2—C4	1.368 (3)	C18—C19	1.354 (4)
N3—C9	1.298 (3)	C18—H18	0.9300
N3—C1	1.461 (3)	C19—C20	1.380 (3)
N4—C14	1.339 (3)	C19—H19	0.9300
N4—C10	1.357 (3)	C20—H20	0.9300
C1—C15	1.500 (3)	C21—C26	1.373 (3)
C1—C2	1.563 (3)	C21—C22	1.393 (3)
C1—H1	0.9800	C22—C23	1.409 (4)
C2—C21	1.509 (3)	C22—H22	0.9300
C2—H2	0.9800	C23—C24	1.342 (4)
C3—C4	1.506 (3)	C23—H23	0.9300
C4—C5	1.359 (3)	C24—C25	1.378 (4)
C5—C6	1.361 (3)	C24—H24	0.9300
C5—H5	0.9300	C25—C26	1.395 (4)
C6—C7	1.373 (4)	C25—H25	0.9300
C6—H6	0.9300	C26—H26	0.9300
C7—C8	1.381 (3)	C28—C27	1.510 (6)
C7—H7	0.9300	C28—H28A	1.0002
C8—H8	0.9300	C28—H28B	0.9388
C9—C10	1.511 (3)	C28—H28C	0.9612
C10—C11	1.372 (3)	C27—O3	1.436 (6)
C11—C12	1.366 (3)	C27—H27A	0.9730
C11—H11	0.9300	C27—H27B	0.9523
C12—C13	1.360 (4)	O3—C27^{i}	1.436 (6)
C12—H12	0.9300		

N3—Ni1—N1	83.26 (8)	C13—C12—H12	120.0
N3—Ni1—N2	166.18 (8)	C11—C12—H12	120.0
N1—Ni1—N2	84.10 (7)	C12—C13—C14	118.6 (2)
N3—Ni1—N4	83.78 (8)	C12—C13—H13	120.7
N1—Ni1—N4	165.74 (8)	C14—C13—H13	120.7
N2—Ni1—N4	109.33 (7)	N4—C14—C13	123.3 (2)
C3—N1—C2	121.62 (17)	N4—C14—H14	118.4
C3—N1—Ni1	119.35 (14)	C13—C14—H14	118.4
C2—N1—Ni1	118.83 (13)	C20—C15—C16	117.7 (2)
C8—N2—C4	116.94 (17)	C20—C15—C1	119.79 (19)
C8—N2—Ni1	131.39 (14)	C16—C15—C1	122.51 (17)
C4—N2—Ni1	111.49 (13)	C17—C16—C15	120.40 (19)
C9—N3—C1	121.56 (15)	C17—C16—H16	119.8
C9—N3—Ni1	119.77 (16)	C15—C16—H16	119.8
C1—N3—Ni1	117.61 (14)	C18—C17—C16	120.5 (2)
C14—N4—C10	116.33 (19)	C18—C17—H17	119.7
C14—N4—Ni1	132.16 (16)	C16—C17—H17	119.7
C10—N4—Ni1	111.49 (13)	C19—C18—C17	119.1 (2)

N3—C1—C15	112.49 (17)	C19—C18—H18	120.4
N3—C1—C2	104.21 (15)	C17—C18—H18	120.4
C15—C1—C2	111.53 (18)	C18—C19—C20	121.1 (2)
N3—C1—H1	109.5	C18—C19—H19	119.5
C15—C1—H1	109.5	C20—C19—H19	119.5
C2—C1—H1	109.5	C19—C20—C15	121.2 (2)
N1—C2—C21	114.66 (17)	C19—C20—H20	119.4
N1—C2—C1	104.85 (16)	C15—C20—H20	119.4
C21—C2—C1	112.35 (18)	C26—C21—C22	116.8 (2)
N1—C2—H2	108.2	C26—C21—C2	124.7 (2)
C21—C2—H2	108.2	C22—C21—C2	118.5 (2)
C1—C2—H2	108.2	C21—C22—C23	122.5 (2)
O1—C3—N1	128.5 (2)	C21—C22—H22	118.8
O1—C3—C4	121.05 (19)	C23—C22—H22	118.8
N1—C3—C4	110.37 (18)	C24—C23—C22	118.0 (3)
C5—C4—N2	121.6 (2)	C24—C23—H23	121.0
C5—C4—C3	124.0 (2)	C22—C23—H23	121.0
N2—C4—C3	114.37 (18)	C23—C24—C25	121.9 (3)
C4—C5—C6	121.3 (2)	C23—C24—H24	119.0
C4—C5—H5	119.4	C25—C24—H24	119.0
C6—C5—H5	119.4	C24—C25—C26	119.2 (3)
C5—C6—C7	117.4 (2)	C24—C25—H25	120.4
C5—C6—H6	121.3	C26—C25—H25	120.4
C7—C6—H6	121.3	C21—C26—C25	121.6 (3)
C6—C7—C8	119.8 (2)	C21—C26—H26	119.2
C6—C7—H7	120.1	C25—C26—H26	119.2
C8—C7—H7	120.1	C27—C28—H28A	108.0
N2—C8—C7	122.8 (2)	C27—C28—H28B	112.0
N2—C8—H8	118.6	H28A—C28—H28B	107.9
C7—C8—H8	118.6	C27—C28—H28C	111.4
O2—C9—N3	128.2 (2)	H28A—C28—H28C	106.1
O2—C9—C10	120.6 (2)	H28B—C28—H28C	111.2
N3—C9—C10	111.19 (18)	O3—C27—C28	135.1 (5)
N4—C10—C11	123.3 (2)	O3—C27—H27A	102.5
N4—C10—C9	113.38 (19)	C28—C27—H27A	103.2
C11—C10—C9	123.2 (2)	O3—C27—H27B	103.6
C12—C11—C10	118.5 (2)	C28—C27—H27B	103.7
C12—C11—H11	120.7	H27A—C27—H27B	106.1
C10—C11—H11	120.7	C27—O3—C27^{i}	114.7 (6)
C13—C12—C11	119.9 (2)		

N3—Ni1—N1—C3	−179.99 (19)	C4—C5—C6—C7	2.1 (4)
N2—Ni1—N1—C3	5.60 (18)	C5—C6—C7—C8	1.0 (4)
N4—Ni1—N1—C3	−155.1 (3)	C4—N2—C8—C7	−1.8 (4)
N3—Ni1—N1—C2	−5.08 (16)	Ni1—N2—C8—C7	−176.47 (19)
N2—Ni1—N1—C2	−179.49 (17)	C6—C7—C8—N2	−1.2 (4)
N4—Ni1—N1—C2	19.8 (4)	C1—N3—C9—O2	8.3 (4)
N3—Ni1—N2—C8	147.3 (3)	Ni1—N3—C9—O2	176.2 (2)

N1—Ni1—N2—C8	171.2 (2)	C1—N3—C9—C10	−171.00 (19)
N4—Ni1—N2—C8	−13.7 (2)	Ni1—N3—C9—C10	−3.1 (3)
N3—Ni1—N2—C4	−27.6 (4)	C14—N4—C10—C11	−0.8 (4)
N1—Ni1—N2—C4	−3.66 (15)	Ni1—N4—C10—C11	177.7 (2)
N4—Ni1—N2—C4	171.40 (15)	C14—N4—C10—C9	174.9 (2)
N1—Ni1—N3—C9	173.64 (18)	Ni1—N4—C10—C9	−6.6 (3)
N2—Ni1—N3—C9	−162.4 (3)	O2—C9—C10—N4	−173.0 (2)
N4—Ni1—N3—C9	−0.38 (18)	N3—C9—C10—N4	6.4 (3)
N1—Ni1—N3—C1	−17.93 (15)	O2—C9—C10—C11	2.8 (4)
N2—Ni1—N3—C1	6.0 (4)	N3—C9—C10—C11	−177.9 (2)
N4—Ni1—N3—C1	168.04 (15)	N4—C10—C11—C12	−0.1 (4)
N3—Ni1—N4—C14	−177.7 (3)	C9—C10—C11—C12	−175.3 (3)
N1—Ni1—N4—C14	157.5 (3)	C10—C11—C12—C13	2.6 (5)
N2—Ni1—N4—C14	−2.2 (3)	C11—C12—C13—C14	−4.2 (5)
N3—Ni1—N4—C10	4.10 (16)	C10—N4—C14—C13	−0.9 (4)
N1—Ni1—N4—C10	−20.7 (4)	Ni1—N4—C14—C13	−179.0 (2)
N2—Ni1—N4—C10	179.62 (16)	C12—C13—C14—N4	3.4 (5)
C9—N3—C1—C15	80.8 (2)	N3—C1—C15—C20	−161.45 (18)
Ni1—N3—C1—C15	−87.42 (17)	C2—C1—C15—C20	81.9 (2)
C9—N3—C1—C2	−158.3 (2)	N3—C1—C15—C16	20.7 (3)
Ni1—N3—C1—C2	33.5 (2)	C2—C1—C15—C16	−96.0 (2)
C3—N1—C2—C21	74.7 (3)	C20—C15—C16—C17	1.0 (3)
Ni1—N1—C2—C21	−100.11 (18)	C1—C15—C16—C17	178.9 (2)
C3—N1—C2—C1	−161.6 (2)	C15—C16—C17—C18	−0.5 (3)
Ni1—N1—C2—C1	23.6 (2)	C16—C17—C18—C19	0.1 (4)
N3—C1—C2—N1	−32.9 (2)	C17—C18—C19—C20	−0.3 (4)
C15—C1—C2—N1	88.7 (2)	C18—C19—C20—C15	0.8 (4)
N3—C1—C2—C21	92.29 (19)	C16—C15—C20—C19	−1.2 (3)
C15—C1—C2—C21	−146.11 (17)	C1—C15—C20—C19	−179.2 (2)
C2—N1—C3—O1	1.8 (4)	N1—C2—C21—C26	6.3 (3)
Ni1—N1—C3—O1	176.5 (2)	C1—C2—C21—C26	−113.2 (3)
C2—N1—C3—C4	179.4 (2)	N1—C2—C21—C22	−173.36 (19)
Ni1—N1—C3—C4	−5.8 (3)	C1—C2—C21—C22	67.0 (2)
C8—N2—C4—C5	5.0 (3)	C26—C21—C22—C23	1.1 (4)
Ni1—N2—C4—C5	−179.3 (2)	C2—C21—C22—C23	−179.2 (2)
C8—N2—C4—C3	−174.1 (2)	C21—C22—C23—C24	−1.2 (4)
Ni1—N2—C4—C3	1.6 (2)	C22—C23—C24—C25	−0.4 (5)
O1—C3—C4—C5	1.2 (4)	C23—C24—C25—C26	2.0 (6)
N1—C3—C4—C5	−176.6 (2)	C22—C21—C26—C25	0.6 (4)
O1—C3—C4—N2	−179.7 (2)	C2—C21—C26—C25	−179.2 (3)
N1—C3—C4—N2	2.4 (3)	C24—C25—C26—C21	−2.1 (5)
N2—C4—C5—C6	−5.3 (4)	C28—C27—O3—C27^{i}	91.6 (7)
C3—C4—C5—C6	173.7 (3)		

Symmetry code: (i) $-x+1, y, -z+2$.