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Diazidobis(5,5'-dimethyl-2,2'-bipyridyl- κ^2N,N')nickel(II) monohydrateJaturong Phatchimkun,^{a*} Palangpon Kongsaree,^b
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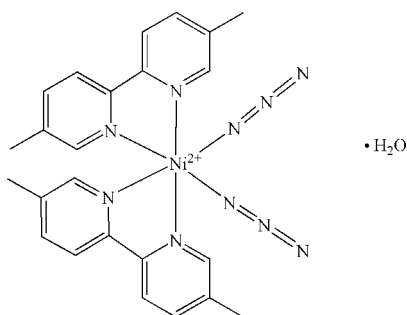
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; H-atom completeness 92%; disorder in solvent or counterion; R factor = 0.043; wR factor = 0.127; data-to-parameter ratio = 21.6.

In the crystal structure of the title compound, $[\text{Ni}(\text{N}_3)_2(\text{C}_{12}\text{H}_{12}\text{N}_2)_2]\cdot\text{H}_2\text{O}$, the Ni^{II} atom is situated on a twofold axis and adopts a distorted octahedral geometry with the two 5,5'-dimethyl-2,2'-bipyridyl (dmbpy) and the two azide ligands in a *cis* arrangement. The water solvent molecule is disordered over two positions in a 1:1 ratio.

Related literature

For general background to 2,2'-bipyridine and its derivatives, see: Blau (1888); Constable (1989); Constable & Steel (1989); Juris *et al.* (1988). For related dmbpy structures, see: van Albada *et al.* (2004, 2005); Catalan *et al.* (1995); Kooijman *et al.* (2002). For Ni–N bond lengths in azido-containing mononuclear nickel(II) complexes, see: Urriaga *et al.* (1995). For an Ni^{II} complex with 5,5'-dimethyl-2,2'-bipyridyl, see: Hou (2008). For a description of the Cambridge Structural Database, see: Allen (2002).



Experimental

Crystal data

$[\text{Ni}(\text{N}_3)_2(\text{C}_{12}\text{H}_{12}\text{N}_2)_2]\cdot\text{H}_2\text{O}$
 $M_r = 529.22$
 Orthorhombic, *Pbcn*
 $a = 17.0770$ (3) Å
 $b = 8.5350$ (5) Å
 $c = 16.6700$ (4) Å

$V = 2429.69$ (16) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.84$ mm⁻¹
 $T = 293$ K
 $0.53 \times 0.45 \times 0.40$ mm

Data collection

Nonius KappaCCD diffractometer
 Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 1996)
 $T_{\text{min}} = 0.630$, $T_{\text{max}} = 0.712$

7908 measured reflections
 4209 independent reflections
 2929 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.127$
 $S = 1.03$
 4209 reflections
 195 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.37$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ni1–N1	2.0882 (13)	Ni1–N3	2.1053 (14)
Ni1–N2	2.0897 (13)		

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

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *COLLECT* and *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *COLLECT*; 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: RN2055).

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Diazidobis(5,5'-dimethyl-2,2'-bipyridyl- κ^2N,N')nickel(II) monohydrate

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Comment

It is 121 years since Blau first reported (Blau, 1888) the synthesis of 2,2'-bipyridine (bpy) and described the first metal complexes of this important ligand. Since then bpy and its derivatives have been continuously and extensively used in several aspects of coordination chemistry (Constable, 1989; Constable & Steel, 1989; Juris *et al.*, 1988). However, up to now comparatively few X-ray crystal structures of ligand 5,5'-dimethyl-2,2'-bipyridyl (dmbpy) have been published (Albada *et al.*, 2004; Catalan *et al.*, 1995; Kooijman *et al.*, 2002). In this study, we report the synthesis and characterization of the new Ni(II) complex containing both dmbpy and azido ligands. Although 52 structures containing dmbpy with metals were found in the Cambridge Structural Database (CSD; Version 5.29, November 2008 update; Allen, 2002) there was only one case (CSD refcode POMFAZ; Hou, 2008) where en is present with both Ni^{II} and 5,5'-dimethyl-2,2'-bipyridyl.

It is found that the Ni ion is coordinated by four nitrogen atoms from dmbpy and two azido nitrogen atoms, taking on a distorted octahedral geometry. The two bidentate ligands have a *cis* disposition around the metal ion, forming practically perpendicular planes [N2—Ni1—N2ⁱ 89.80 (7), N1—Ni1—N1ⁱ 176.19 (7)°] (symmetry codes: *i* -*x* + 2, *y*, -*z* + 1/2). The rigidity of these ligands causes the bond angles N1—Ni1—N2, 78.26 (5)° to deviate significantly from orthogonality. This causes the geometry about the Ni^{II} ion to deviate slightly from that of an ideal octahedron. The Ni(1)—N(dmbpy) bond distances in a related complex [2.0897 (13) and 2.0882 (13) Å] (Urutiaga *et al.*, 1995) are almost the same as those found in the Ni(II) compound of [Ni^{II}(bpy)₂(N₃)₂] [2.067 (2)–2.114 (2) Å]. Good agreement is observed between the Ni(1)—N(azido) bond distance of 2.1053 (14) Å and those reported [2.094 (2) and 2.102 (3) Å] (Urutiaga *et al.*, 1995) for azido containing mononuclear nickel(II) complexes.

Experimental

All chemicals were obtained commercially and used without further purification. The compound was prepared by adding a warm solution of dmbpy (0.181 g, 1.0 mmol) in methanol (15 ml) to a warming aqueous solution (10 ml) of Ni(CH₃COO)₂·4H₂O (0.123 g, 0.5 mmol). Afterward a solid of NaN₃ (0.065 g, 1.0 mmol) was added. The green solution was slowly evaporated at room temperature and after few days, green plate-shaped crystals of [Ni(dmbpy)₂(N₃)₂].H₂O formed. They were filtered off, washed with mother liquid and air-dried. Yield *ca* 89%. Elemental analysis (%) found (calculated): C 54.4 (54.6), H 4.9 (4.8), N 26.3 (26.5). The IR spectrum shows the band corresponding to the asymmetric stretch of the azido ion, $\nu_{\text{as}}(\text{N}_3)$, split at 2072 and 2024 cm⁻¹. This indicates that the azido ligand is bonded asymmetrically by its two terminal N atoms.

Refinement

The water O atom is disordered which site occupancies of 0.5 and 0.5. All non-H atoms were refined anisotropically. H atoms in aromatic were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances of

supplementary materials

0.96–1.01 Å [U_{iso} (H) = 1.2U_{eq} (C)]. H atoms of methyl groups of dmbpy were placed in calculated positions and refined with a riding model C—H = 0.96 Å.

Figures

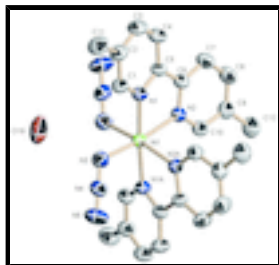


Fig. 1. A view of the title structure with the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted for clarity.

Diazidobis(5,5'-dimethyl-2,2'-bipyridyl- κ^2N,N')nickel(II) monohydrate

Crystal data

$[\text{Ni}(\text{N}_3)_2(\text{C}_{12}\text{H}_{12}\text{N}_2)_2] \cdot \text{H}_2\text{O}$

$M_r = 529.22$

Orthorhombic, $Pbcn$

Hall symbol: $-P\ 2n\ 2ab$

$a = 17.0770$ (3) Å

$b = 8.5350$ (5) Å

$c = 16.6700$ (4) Å

$V = 2429.69$ (16) Å³

$Z = 4$

$F_{000} = 1096$

$D_x = 1.444$ Mg m⁻³

$D_m = 1.440$ Mg m⁻³

D_m measured by flotation in aqueous KI

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7908 reflections

$\theta = 2.7$ – 27.5°

$\mu = 0.84$ mm⁻¹

$T = 293$ K

Plate, green

$0.53 \times 0.45 \times 0.40$ mm

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ K

ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\text{min}} = 0.630$, $T_{\text{max}} = 0.712$

7908 measured reflections

4209 independent reflections

2929 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\text{max}} = 32.0^\circ$

$\theta_{\text{min}} = 2.7^\circ$

$h = -25 \rightarrow 25$

$k = -12 \rightarrow 12$

$l = -24 \rightarrow 24$

Refinement

Refinement on F^2

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of

	independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.043$	$w = 1/[\sigma^2(F_o^2) + (0.0689P)^2 + 0.3234P]$
$wR(F^2) = 0.127$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{\max} < 0.001$
4209 reflections	$\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$
195 parameters	$\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.023 (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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Ni1	1.0000	0.25758 (3)	0.2500	0.03045 (11)	
C1	0.85037 (9)	0.1839 (2)	0.15504 (10)	0.0394 (4)	
C2	0.77052 (9)	0.1813 (2)	0.13931 (10)	0.0430 (4)	
C3	0.72270 (11)	0.2632 (2)	0.19237 (13)	0.0485 (5)	
C4	0.75485 (10)	0.3478 (3)	0.25456 (10)	0.0444 (4)	
C5	0.83592 (9)	0.3501 (2)	0.26388 (9)	0.0351 (3)	
C6	0.87691 (8)	0.44736 (19)	0.32433 (9)	0.0346 (3)	
C7	0.83951 (10)	0.5554 (2)	0.37348 (11)	0.0455 (4)	
C8	0.88410 (10)	0.6520 (2)	0.42157 (11)	0.0474 (4)	
C9	0.96489 (10)	0.6414 (2)	0.42151 (9)	0.0404 (4)	
C10	0.99748 (9)	0.5262 (2)	0.37327 (10)	0.0378 (3)	
C11	0.73848 (11)	0.0947 (3)	0.06809 (12)	0.0571 (5)	
C12	1.01596 (15)	0.7508 (2)	0.46878 (14)	0.0526 (5)	
N1	0.88262 (7)	0.26572 (15)	0.21515 (8)	0.0341 (3)	
N2	0.95560 (7)	0.43102 (16)	0.32590 (7)	0.0339 (3)	
N3	1.02626 (9)	0.08562 (18)	0.16354 (8)	0.0434 (3)	
N4	1.08684 (8)	0.07905 (18)	0.12842 (8)	0.0402 (3)	
N5	1.14499 (9)	0.0716 (3)	0.09306 (11)	0.0655 (5)	
O19	0.9708 (3)	0.7988 (4)	0.2398 (3)	0.0853 (15)	0.50
H1	0.8876 (10)	0.120 (2)	0.1214 (11)	0.044 (5)*	
H3	0.6660 (14)	0.261 (2)	0.1832 (13)	0.053 (6)*	

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H4	0.7209 (11)	0.404 (2)	0.2906 (12)	0.053 (6)*
H7	0.7824 (12)	0.563 (2)	0.3731 (11)	0.049 (5)*
H8	0.8591 (13)	0.735 (2)	0.4557 (14)	0.054 (6)*
H10	1.0548 (12)	0.509 (2)	0.3715 (11)	0.045 (5)*
H11A	0.7797	0.0357	0.0432	0.086*
H11B	0.7175	0.1683	0.0302	0.086*
H11C	0.6978	0.0247	0.0853	0.086*
H12A	1.0385	0.8272	0.4334	0.079*
H12B	1.0570	0.6922	0.4944	0.079*
H12C	0.9850	0.8028	0.5088	0.079*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02557 (16)	0.03435 (17)	0.03144 (16)	0.000	0.00161 (9)	0.000
C1	0.0336 (7)	0.0443 (9)	0.0403 (8)	-0.0033 (7)	-0.0006 (6)	-0.0012 (7)
C2	0.0361 (8)	0.0476 (10)	0.0455 (8)	-0.0063 (7)	-0.0043 (6)	0.0019 (8)
C3	0.0302 (8)	0.0605 (12)	0.0549 (10)	-0.0022 (7)	-0.0036 (7)	0.0015 (8)
C4	0.0294 (7)	0.0546 (11)	0.0492 (9)	0.0034 (7)	0.0021 (6)	-0.0002 (8)
C5	0.0292 (7)	0.0377 (8)	0.0384 (7)	-0.0002 (6)	0.0018 (5)	0.0033 (6)
C6	0.0304 (7)	0.0378 (8)	0.0354 (7)	0.0030 (6)	0.0020 (5)	0.0024 (6)
C7	0.0369 (8)	0.0533 (11)	0.0462 (9)	0.0066 (7)	0.0046 (7)	-0.0081 (8)
C8	0.0483 (9)	0.0506 (11)	0.0433 (9)	0.0086 (8)	0.0059 (7)	-0.0100 (8)
C9	0.0477 (9)	0.0399 (9)	0.0336 (7)	-0.0002 (7)	-0.0016 (6)	-0.0010 (7)
C10	0.0342 (7)	0.0411 (8)	0.0381 (8)	-0.0004 (6)	-0.0013 (5)	-0.0009 (7)
C11	0.0462 (10)	0.0693 (14)	0.0556 (11)	-0.0115 (9)	-0.0091 (8)	-0.0088 (10)
C12	0.0645 (12)	0.0489 (11)	0.0444 (10)	-0.0032 (8)	-0.0044 (9)	-0.0116 (8)
N1	0.0281 (6)	0.0384 (7)	0.0360 (7)	-0.0014 (5)	0.0007 (5)	0.0000 (5)
N2	0.0313 (6)	0.0357 (7)	0.0347 (6)	0.0013 (5)	0.0017 (4)	-0.0005 (5)
N3	0.0424 (7)	0.0447 (8)	0.0431 (7)	-0.0017 (6)	0.0084 (6)	-0.0076 (6)
N4	0.0373 (7)	0.0448 (8)	0.0383 (7)	0.0076 (6)	-0.0032 (5)	-0.0040 (6)
N5	0.0386 (8)	0.0938 (15)	0.0640 (10)	0.0137 (9)	0.0085 (7)	-0.0099 (10)
O19	0.132 (5)	0.0521 (16)	0.072 (3)	-0.021 (2)	0.026 (3)	-0.0070 (18)

Geometric parameters (\AA , $^\circ$)

Ni1—Ni1 ⁱ	2.0882 (13)	C6—C7	1.389 (2)
Ni1—N1	2.0882 (13)	C7—C8	1.379 (3)
Ni1—N2 ⁱ	2.0897 (13)	C7—H7	0.978 (19)
Ni1—N2	2.0897 (13)	C8—C9	1.383 (2)
Ni1—N3	2.1053 (14)	C8—H8	1.00 (2)
Ni1—N3 ⁱ	2.1053 (14)	C9—C10	1.387 (2)
C1—N1	1.340 (2)	C9—C12	1.501 (3)
C1—C2	1.389 (2)	C10—N2	1.340 (2)
C1—H1	1.008 (19)	C10—H10	0.99 (2)
C2—C3	1.392 (3)	C11—H11A	0.9600
C2—C11	1.502 (3)	C11—H11B	0.9600
C3—C4	1.377 (3)	C11—H11C	0.9600

C3—H3	0.98 (2)	C12—H12A	0.9600
C4—C5	1.393 (2)	C12—H12B	0.9600
C4—H4	0.96 (2)	C12—H12C	0.9600
C5—N1	1.347 (2)	N3—N4	1.1901 (19)
C5—C6	1.481 (2)	N4—N5	1.157 (2)
C6—N2	1.3512 (18)	O19—O19 ⁱ	1.053 (9)
N1 ⁱ —Ni1—N1	176.19 (7)	C8—C7—C6	119.07 (15)
N1 ⁱ —Ni1—N2 ⁱ	78.26 (5)	C8—C7—H7	121.0 (12)
N1—Ni1—N2 ⁱ	98.99 (5)	C6—C7—H7	119.9 (12)
N1 ⁱ —Ni1—N2	98.99 (5)	C7—C8—C9	120.74 (16)
N1—Ni1—N2	78.26 (5)	C7—C8—H8	121.0 (13)
N2 ⁱ —Ni1—N2	89.80 (7)	C9—C8—H8	118.2 (13)
N1 ⁱ —Ni1—N3	90.53 (5)	C8—C9—C10	116.59 (16)
N1—Ni1—N3	92.13 (5)	C8—C9—C12	122.57 (17)
N2 ⁱ —Ni1—N3	90.12 (6)	C10—C9—C12	120.81 (17)
N2—Ni1—N3	170.26 (5)	N2—C10—C9	123.88 (15)
N1 ⁱ —Ni1—N3 ⁱ	92.13 (5)	N2—C10—H10	114.9 (11)
N1—Ni1—N3 ⁱ	90.53 (5)	C9—C10—H10	121.2 (11)
N2 ⁱ —Ni1—N3 ⁱ	170.26 (5)	C2—C11—H11A	109.5
N2—Ni1—N3 ⁱ	90.12 (6)	C2—C11—H11B	109.5
N3—Ni1—N3 ⁱ	91.61 (8)	H11A—C11—H11B	109.5
N1—C1—C2	123.57 (16)	C2—C11—H11C	109.5
N1—C1—H1	116.1 (10)	H11A—C11—H11C	109.5
C2—C1—H1	120.3 (10)	H11B—C11—H11C	109.5
C1—C2—C3	116.64 (16)	C9—C12—H12A	109.5
C1—C2—C11	120.97 (17)	C9—C12—H12B	109.5
C3—C2—C11	122.39 (16)	H12A—C12—H12B	109.5
C4—C3—C2	120.49 (16)	C9—C12—H12C	109.5
C4—C3—H3	121.3 (12)	H12A—C12—H12C	109.5
C2—C3—H3	118.2 (12)	H12B—C12—H12C	109.5
C3—C4—C5	119.18 (17)	C1—N1—C5	119.11 (14)
C3—C4—H4	119.4 (12)	C1—N1—Ni1	125.82 (11)
C5—C4—H4	121.4 (12)	C5—N1—Ni1	114.73 (10)
N1—C5—C4	120.87 (15)	C10—N2—C6	118.67 (13)
N1—C5—C6	115.47 (13)	C10—N2—Ni1	126.35 (10)
C4—C5—C6	123.61 (15)	C6—N2—Ni1	114.97 (10)
N2—C6—C7	120.95 (15)	N4—N3—Ni1	123.70 (12)
N2—C6—C5	115.17 (13)	N5—N4—N3	178.73 (18)
C7—C6—C5	123.77 (14)		
N1—C1—C2—C3	-3.3 (3)	N2—Ni1—N1—C1	176.04 (14)
N1—C1—C2—C11	176.43 (18)	N3—Ni1—N1—C1	-2.32 (14)
C1—C2—C3—C4	3.1 (3)	N3 ⁱ —Ni1—N1—C1	-93.95 (14)
C11—C2—C3—C4	-176.62 (19)	N2 ⁱ —Ni1—N1—C5	-98.70 (11)
C2—C3—C4—C5	-0.2 (3)	N2—Ni1—N1—C5	-10.79 (11)
C3—C4—C5—N1	-2.9 (3)	N3—Ni1—N1—C5	170.84 (11)

supplementary materials

C3—C4—C5—C6	174.78 (17)	N3 ⁱ —Ni1—N1—C5	79.22 (12)
N1—C5—C6—N2	-4.0 (2)	C9—C10—N2—C6	0.2 (2)
C4—C5—C6—N2	178.28 (16)	C9—C10—N2—Ni1	178.82 (12)
N1—C5—C6—C7	172.35 (16)	C7—C6—N2—C10	-3.0 (2)
C4—C5—C6—C7	-5.4 (3)	C5—C6—N2—C10	173.45 (14)
N2—C6—C7—C8	3.1 (3)	C7—C6—N2—Ni1	178.23 (13)
C5—C6—C7—C8	-173.01 (16)	C5—C6—N2—Ni1	-5.34 (17)
C6—C7—C8—C9	-0.4 (3)	N1 ⁱ —Ni1—N2—C10	7.22 (14)
C7—C8—C9—C10	-2.2 (3)	N1—Ni1—N2—C10	-170.09 (14)
C7—C8—C9—C12	175.99 (19)	N2 ⁱ —Ni1—N2—C10	-70.86 (13)
C8—C9—C10—N2	2.4 (3)	N3 ⁱ —Ni1—N2—C10	99.40 (14)
C12—C9—C10—N2	-175.83 (16)	N1 ⁱ —Ni1—N2—C6	-174.10 (10)
C2—C1—N1—C5	0.4 (3)	N1—Ni1—N2—C6	8.59 (10)
C2—C1—N1—Ni1	173.26 (13)	N2 ⁱ —Ni1—N2—C6	107.82 (11)
C4—C5—N1—C1	2.8 (2)	N3 ⁱ —Ni1—N2—C6	-81.92 (11)
C6—C5—N1—C1	-175.05 (14)	N1 ⁱ —Ni1—N3—N4	-32.56 (15)
C4—C5—N1—Ni1	-170.89 (13)	N1—Ni1—N3—N4	144.70 (14)
C6—C5—N1—Ni1	11.29 (17)	N2 ⁱ —Ni1—N3—N4	45.70 (14)
N2 ⁱ —Ni1—N1—C1	88.14 (14)	N3 ⁱ —Ni1—N3—N4	-124.71 (16)

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

Fig. 1

