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trans-Bis(*N*′-isopropylidenebenzohydrazidato-κ^2N',O)bis(pyridine-κN)nickel(II)

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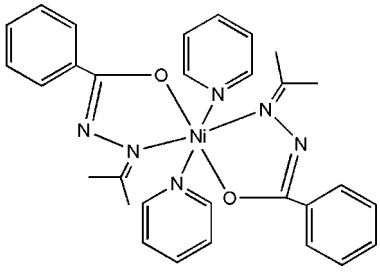
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.028; wR factor = 0.078; data-to-parameter ratio = 14.1.

The complex molecule of the title compound, $[Ni(C_{10}H_{11}N_2O)_2(C_5H_5N)_2]$, has a crystallographically imposed centre of symmetry. The Ni^{II} atom is coordinated in a distorted octahedral geometry by the O and N atoms of two *trans* arranged anionic bidentate hydrazone ligands forming the equatorial plane and by the N atoms of two pyridine molecules at the axial positions. In the crystal, intermolecular C—H···N hydrogen bonds link the molecules into columns parallel to the *b* axis.

Related literature

For the biological and coordination properties of aroylhydrazones, see: Ali *et al.* (2004); Carcelli *et al.* (1995); Cheng *et al.* (1996); Zhang *et al.* (2011).



Experimental

Crystal data

$[Ni(C_{10}H_{11}N_2O)_2(C_5H_5N)_2]$
M_r = 567.33
Monoclinic, $C2/c$
a = 15.419 (5) Å
b = 9.242 (3) Å
c = 21.295 (9) Å
β = 109.924 (5)°
V = 2853.1 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.72 mm^{-1}
T = 298 K
0.23 × 0.14 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.852, T_{max} = 0.919
7093 measured reflections
2514 independent reflections
2285 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.078
S = 1.10
2514 reflections
178 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···N1^{i}	0.93	2.50	3.382 (3)	157

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

Data collection: *SMART* (Bruker, 1996); cell refinement: *SAINT* (Bruker, 1996); 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: RZ2613).

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supporting information

Acta Cryst. (2011). E**67**, m978 [doi:10.1107/S1600536811024214]

trans-Bis(*N*′-isopropylidenebenzohydrazidato-κ^2N',O)bis(pyridine-κN)nickel(II)

Chang-Zheng Zheng, Liang Wang and Juan Liu

S1. Comment

Hydrazones are an important class of Schiff bases compounds which has attracted much attention because of their biological activities (Carcelli *et al.*, 1995), chemical and industrial versatility, and strong tendency to chelate to transition metals (Zhang *et al.*, 2011; Ali *et al.*, 2004; Cheng *et al.*, 1996). As an extension of our work on the structural characterization of aroylhydrazone derivatives, the title compound was synthesized and its crystal structure is reported here.

In the title compound, the complex molecule has crystallographically imposed centre of symmetry (Fig. 1). The coordination polyhedron about the nickel metal is distorted octahedral, with the N, O atoms of two *trans*-arranged anionic bidentate hydrazone ligands at the equatorial plane and by the N atoms of two pyridine molecules occupying the axial positions. In the crystal structure, complex molecules are linked by intermolecular C—H···N hydrogen bonds (Table 1) into columns parallel to the *b* axis.

S2. Experimental

Ethyl benzoate (6.00 g, 0.04 mol) was dissolved in ethnol (30 ml) at room temperature and heated at 363 K, followed by the addition of hydrazine hydrate (2.40 g, 0.048 mol). Subsequently, the mixture was refluxed for 9 h, and then cooled to room temperature. The crystals precipitated were collected by filtration. The product was recrystallized from ethanol and dried under reduced pressure to give benzoylhydrazine. Benzoylhydrazine (3.40 g, 0.025 mol) was dissolved in ethanol (20 ml) at room temperature and heated at 363 K, followed by the addition of dimethyl ketone (1.45 g, 0.025 mol). Subsequently, the mixture was refluxed for 10 h, and then cooled to room temperature. The solid phase precipitated was collected by filtration. The product was recrystallized from ethanol and dried under reduced pressure to give *N*′-[(*E*)- dimethylketone]-benzohydrazide. A mixture of *N*′-[(*E*)-dimethylketone]-benzohydrazide (0.018 g, 0.10 mmol), $NiC_{l2}.6H_2O$ (0.024 g, 0.10 mmol), pyridine (0.0079 g, 0.10 mmol), H_2O (5.00 ml) and several drop of methanol was placed in a Parr Teflon-lined stainless steel vessel (25 ml), and then the vessel was sealed and heated at 393 K for 3 d. After the mixture was slowly cooled to room temperature, red crystals suitable for X-ray analysis were obtained (yield 37%).

S3. Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms,with C—H = 0.93-0.96 Å, and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for methyl H atoms.

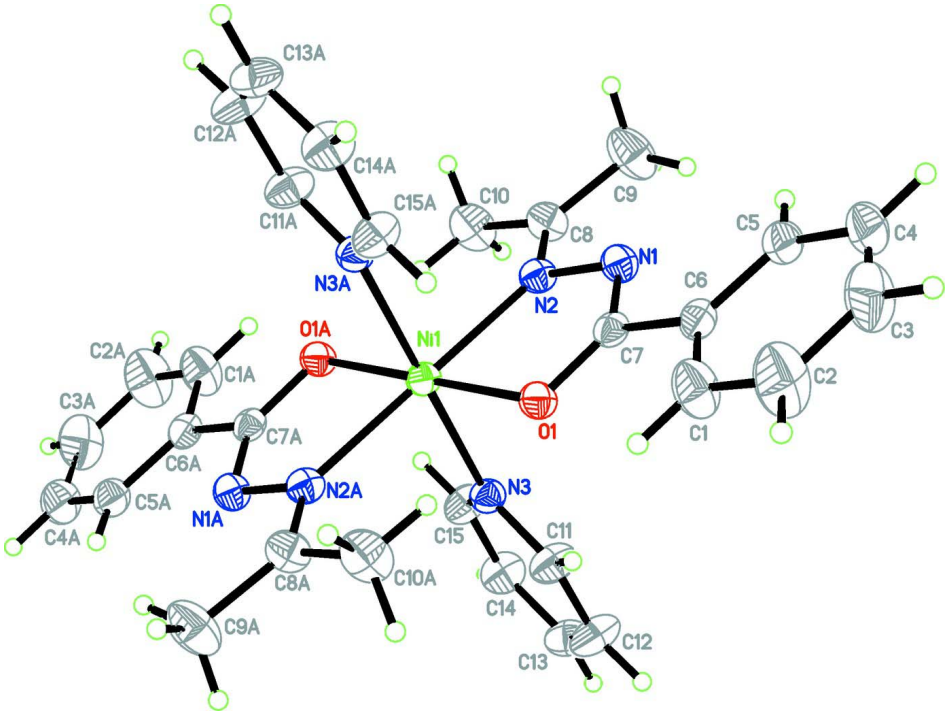

Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level. H atoms are presented as small spheres of arbitrary radius. Symmetry code: (A) -x, 1-y, -z.

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Crystal data

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Monoclinic, $C2/c$
Hall symbol: -C 2yc
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$b = 9.242\ (3)$ Å
$c = 21.295\ (9)$ Å
$\beta = 109.924\ (5)°$
$V = 2853.1\ (18)$ Å^3
$Z = 4$
$F(000) = 1192$
$D_x = 1.321$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5423 reflections
$\theta = 2.6–28.5°$
$\mu = 0.72$ mm^{-1}
$T = 298$ K
Block, red
0.23 × 0.14 × 0.12 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)
$T_{min} = 0.852$, $T_{max} = 0.919$
7093 measured reflections
2514 independent reflections
2285 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 25.1°$, $\theta_{min} = 2.0°$
$h = -18\rightarrow16$
$k = -10\rightarrow8$
$l = -24\rightarrow25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.078$
$S = 1.10$
2514 reflections
178 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 2.5175P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.0113 (15)

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}
Ni1	0.0000	0.5000	0.0000	0.03337 (12)
O1	0.12440 (8)	0.44811 (14)	0.06671 (6)	0.0385 (3)
N1	0.04126 (10)	0.34430 (16)	0.12653 (8)	0.0393 (4)
N2	−0.03583 (10)	0.40836 (16)	0.07882 (7)	0.0375 (3)
N3	0.00804 (10)	0.71169 (16)	0.04278 (7)	0.0383 (3)
C1	0.28411 (15)	0.3172 (3)	0.14883 (12)	0.0636 (6)
H1	0.2865	0.3759	0.1140	0.076*
C2	0.36373 (18)	0.2488 (4)	0.18906 (15)	0.0871 (9)
H2	0.4190	0.2619	0.1812	0.104*
C3	0.36080 (19)	0.1624 (3)	0.23998 (14)	0.0788 (8)
H3	0.4138	0.1154	0.2668	0.095*
C4	0.27956 (18)	0.1450 (3)	0.25151 (12)	0.0665 (7)
H4	0.2775	0.0861	0.2864	0.080*
C5	0.20085 (15)	0.2138 (2)	0.21199 (10)	0.0504 (5)
H5	0.1462	0.2020	0.2208	0.061*
C6	0.20201 (13)	0.2999 (2)	0.15964 (9)	0.0397 (4)
C7	0.11672 (12)	0.37097 (18)	0.11420 (9)	0.0348 (4)
C8	−0.11104 (14)	0.3927 (2)	0.09097 (10)	0.0474 (5)
C9	−0.11699 (18)	0.3144 (3)	0.15068 (13)	0.0741 (8)
H9A	−0.1454	0.2217	0.1373	0.111*
H9B	−0.1534	0.3699	0.1707	0.111*
H9C	−0.0561	0.3012	0.1825	0.111*
C10	−0.19837 (15)	0.4545 (3)	0.04456 (13)	0.0654 (6)

H10A	−0.1871	0.4978	0.0071	0.098*
H10B	−0.2210	0.5267	0.0675	0.098*
H10C	−0.2434	0.3790	0.0291	0.098*
C11	0.08853 (14)	0.7625 (2)	0.08221 (11)	0.0529 (5)
H11	0.1406	0.7046	0.0902	0.063*
C12	0.09888 (16)	0.8958 (2)	0.11177 (13)	0.0646 (6)
H12	0.1566	0.9265	0.1397	0.078*
C13	0.02369 (17)	0.9830 (2)	0.09987 (12)	0.0551 (6)
H13	0.0289	1.0742	0.1193	0.066*
C14	−0.05893 (15)	0.9331 (2)	0.05897 (12)	0.0554 (5)
H14	−0.1115	0.9902	0.0496	0.066*
C15	−0.06440 (14)	0.7977 (2)	0.03149 (11)	0.0497 (5)
H15	−0.1216	0.7648	0.0037	0.060*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02844 (18)	0.03087 (18)	0.03696 (19)	0.00162 (12)	0.00618 (13)	0.00258 (13)
O1	0.0330 (6)	0.0372 (7)	0.0409 (7)	0.0014 (5)	0.0068 (5)	0.0065 (6)
N1	0.0378 (8)	0.0370 (8)	0.0406 (8)	0.0051 (7)	0.0101 (7)	0.0043 (7)
N2	0.0345 (8)	0.0331 (8)	0.0427 (8)	0.0019 (6)	0.0106 (7)	0.0009 (6)
N3	0.0373 (8)	0.0346 (8)	0.0404 (8)	−0.0003 (7)	0.0099 (7)	0.0009 (6)
C1	0.0458 (12)	0.0827 (17)	0.0591 (14)	0.0196 (12)	0.0138 (10)	0.0214 (12)
C2	0.0473 (14)	0.126 (3)	0.0819 (19)	0.0321 (16)	0.0147 (13)	0.0277 (19)
C3	0.0627 (16)	0.0872 (19)	0.0689 (17)	0.0342 (14)	−0.0003 (13)	0.0178 (14)
C4	0.0732 (17)	0.0562 (14)	0.0525 (13)	0.0089 (12)	−0.0014 (12)	0.0165 (11)
C5	0.0527 (12)	0.0444 (11)	0.0458 (11)	0.0008 (10)	0.0061 (9)	0.0033 (9)
C6	0.0409 (10)	0.0333 (9)	0.0375 (10)	0.0046 (8)	0.0039 (8)	−0.0028 (7)
C7	0.0371 (9)	0.0268 (9)	0.0354 (9)	0.0020 (7)	0.0059 (7)	−0.0035 (7)
C8	0.0410 (10)	0.0492 (11)	0.0549 (12)	0.0033 (9)	0.0201 (9)	0.0051 (9)
C9	0.0614 (15)	0.094 (2)	0.0782 (17)	0.0140 (14)	0.0386 (13)	0.0308 (15)
C10	0.0394 (11)	0.0851 (17)	0.0734 (16)	0.0077 (12)	0.0213 (11)	0.0201 (14)
C11	0.0404 (11)	0.0412 (11)	0.0693 (14)	0.0015 (9)	0.0087 (10)	−0.0077 (10)
C12	0.0497 (13)	0.0499 (13)	0.0834 (17)	−0.0085 (10)	0.0086 (12)	−0.0191 (12)
C13	0.0625 (14)	0.0358 (11)	0.0698 (15)	−0.0047 (10)	0.0261 (12)	−0.0104 (10)
C14	0.0507 (12)	0.0463 (12)	0.0691 (14)	0.0106 (10)	0.0204 (11)	−0.0057 (11)
C15	0.0387 (10)	0.0480 (11)	0.0577 (12)	0.0028 (9)	0.0104 (9)	−0.0089 (10)

Geometric parameters (Å, º)

Ni1—O1^{i}	2.0175 (13)	C5—C6	1.374 (3)
Ni1—O1	2.0175 (13)	C5—H5	0.9300
Ni1—N2	2.1148 (16)	C6—C7	1.493 (2)
Ni1—N2^{i}	2.1148 (16)	C8—C10	1.486 (3)
Ni1—N3	2.1439 (16)	C8—C9	1.493 (3)
Ni1—N3^{i}	2.1439 (16)	C9—H9A	0.9600
O1—C7	1.275 (2)	C9—H9B	0.9600
N1—C7	1.301 (2)	C9—H9C	0.9600

N1—N2	1.404 (2)	C10—H10A	0.9600
N2—C8	1.279 (2)	C10—H10B	0.9600
N3—C15	1.325 (2)	C10—H10C	0.9600
N3—C11	1.325 (2)	C11—C12	1.368 (3)
C1—C6	1.371 (3)	C11—H11	0.9300
C1—C2	1.387 (3)	C12—C13	1.363 (3)
C1—H1	0.9300	C12—H12	0.9300
C2—C3	1.360 (4)	C13—C14	1.356 (3)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.366 (4)	C14—C15	1.372 (3)
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.375 (3)	C15—H15	0.9300
C4—H4	0.9300		

$O1^{i}$—Ni1—O1	180.00 (9)	C4—C5—H5	119.6
$O1^{i}$—Ni1—N2	102.19 (6)	C1—C6—C5	118.16 (19)
O1—Ni1—N2	77.81 (6)	C1—C6—C7	119.95 (18)
$O1^{i}$—Ni1—$N2^{i}$	77.81 (6)	C5—C6—C7	121.87 (18)
O1—Ni1—$N2^{i}$	102.19 (6)	O1—C7—N1	126.67 (16)
N2—Ni1—$N2^{i}$	180.00 (8)	O1—C7—C6	117.41 (16)
$O1^{i}$—Ni1—N3	89.18 (6)	N1—C7—C6	115.89 (16)
O1—Ni1—N3	90.82 (6)	N2—C8—C10	120.02 (18)
N2—Ni1—N3	91.21 (6)	N2—C8—C9	123.37 (19)
$N2^{i}$—Ni1—N3	88.79 (6)	C10—C8—C9	116.61 (18)
$O1^{i}$—Ni1—$N3^{i}$	90.82 (6)	C8—C9—H9A	109.5
O1—Ni1—$N3^{i}$	89.18 (6)	C8—C9—H9B	109.5
N2—Ni1—$N3^{i}$	88.79 (6)	H9A—C9—H9B	109.5
$N2^{i}$—Ni1—$N3^{i}$	91.21 (6)	C8—C9—H9C	109.5
N3—Ni1—$N3^{i}$	180.00 (8)	H9A—C9—H9C	109.5
C7—O1—Ni1	111.61 (11)	H9B—C9—H9C	109.5
C7—N1—N2	111.65 (14)	C8—C10—H10A	109.5
C8—N2—N1	114.09 (16)	C8—C10—H10B	109.5
C8—N2—Ni1	134.88 (14)	H10A—C10—H10B	109.5
N1—N2—Ni1	110.93 (11)	C8—C10—H10C	109.5
C15—N3—C11	116.89 (17)	H10A—C10—H10C	109.5
C15—N3—Ni1	123.21 (13)	H10B—C10—H10C	109.5
C11—N3—Ni1	119.90 (13)	N3—C11—C12	123.2 (2)
C6—C1—C2	121.1 (2)	N3—C11—H11	118.4
C6—C1—H1	119.5	C12—C11—H11	118.4
C2—C1—H1	119.5	C13—C12—C11	119.3 (2)
C3—C2—C1	119.8 (3)	C13—C12—H12	120.4
C3—C2—H2	120.1	C11—C12—H12	120.4
C1—C2—H2	120.1	C14—C13—C12	118.2 (2)
C2—C3—C4	119.6 (2)	C14—C13—H13	120.9
C2—C3—H3	120.2	C12—C13—H13	120.9
C4—C3—H3	120.2	C13—C14—C15	119.5 (2)
C3—C4—C5	120.5 (2)	C13—C14—H14	120.3
C3—C4—H4	119.7	C15—C14—H14	120.3

C5—C4—H4	119.7	N3—C15—C14	123.00 (19)
C6—C5—C4	120.8 (2)	N3—C15—H15	118.5
C6—C5—H5	119.6	C14—C15—H15	118.5
N2—Ni1—O1—C7	9.82 (11)	C3—C4—C5—C6	0.8 (4)
N2^{i}—Ni1—O1—C7	−170.18 (11)	C2—C1—C6—C5	0.7 (4)
N3—Ni1—O1—C7	100.88 (12)	C2—C1—C6—C7	−178.0 (2)
N3^{i}—Ni1—O1—C7	−79.12 (12)	C4—C5—C6—C1	−1.2 (3)
C7—N1—N2—C8	−175.97 (16)	C4—C5—C6—C7	177.42 (19)
C7—N1—N2—Ni1	7.01 (17)	Ni1—O1—C7—N1	−10.2 (2)
O1^{i}—Ni1—N2—C8	−5.3 (2)	Ni1—O1—C7—C6	168.00 (12)
O1—Ni1—N2—C8	174.7 (2)	N2—N1—C7—O1	1.9 (2)
N3—Ni1—N2—C8	84.12 (19)	N2—N1—C7—C6	−176.30 (14)
N3^{i}—Ni1—N2—C8	−95.88 (19)	C1—C6—C7—O1	−0.9 (3)
O1^{i}—Ni1—N2—N1	170.85 (10)	C5—C6—C7—O1	−179.55 (17)
O1—Ni1—N2—N1	−9.15 (10)	C1—C6—C7—N1	177.46 (19)
N3—Ni1—N2—N1	−99.72 (11)	C5—C6—C7—N1	−1.2 (3)
N3^{i}—Ni1—N2—N1	80.28 (11)	N1—N2—C8—C10	−179.78 (19)
O1^{i}—Ni1—N3—C15	16.25 (16)	Ni1—N2—C8—C10	−3.7 (3)
O1—Ni1—N3—C15	−163.75 (16)	N1—N2—C8—C9	0.4 (3)
N2—Ni1—N3—C15	−85.93 (16)	Ni1—N2—C8—C9	176.48 (18)
N2^{i}—Ni1—N3—C15	94.07 (16)	C15—N3—C11—C12	1.2 (3)
O1^{i}—Ni1—N3—C11	−163.18 (16)	Ni1—N3—C11—C12	−179.37 (19)
O1—Ni1—N3—C11	16.82 (16)	N3—C11—C12—C13	−1.0 (4)
N2—Ni1—N3—C11	94.64 (16)	C11—C12—C13—C14	0.2 (4)
N2^{i}—Ni1—N3—C11	−85.36 (16)	C12—C13—C14—C15	0.4 (4)
C6—C1—C2—C3	0.3 (5)	C11—N3—C15—C14	−0.5 (3)
C1—C2—C3—C4	−0.7 (5)	Ni1—N3—C15—C14	−179.99 (17)
C2—C3—C4—C5	0.1 (4)	C13—C14—C15—N3	−0.2 (4)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···N1^{ii}	0.93	2.50	3.382 (3)	157

Symmetry code: (ii) $x, y+1, z$.