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Bis(dimethylformamide-2κO)bis[μ-1-(2-oxidobenzoyl)-2-(phenoxyacetyl)-hydrazine(3−)]-1$\kappa^3 O,N,O'$:2$\kappa^2 N,O''$;-2$\kappa^2 N,O''$:3$\kappa^3 O,N,O'$-dipyridine-1κN,3κN-trinickel(II)

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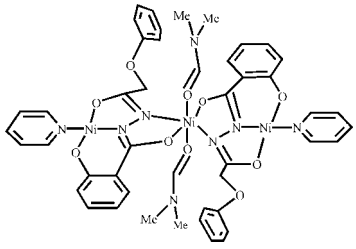
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; R factor = 0.060; wR factor = 0.129; data-to-parameter ratio = 17.2.

The trinuclear nickel(II) title compound, $[Ni_3(C_{15}H_{12}N_2O_4)_2(C_5H_5N)_2(C_3H_6NO)_2]$, possesses a crystallographically imposed centre of symmetry. The central Ni atom adopts an axially elongated octahedral geometry, involving two N and two O atoms from two phenoxyacetylsalicylhydrazidate ligands in the equatorial plane and two O atoms from two dimethylformamide molecules in the axial positions. The inversion-related outer Ni atoms have square-planar coordination environments provided by two O atoms and one N atom of a phenoxyacetylsalicylhydrazidate ligand and by the N atom of a pyridine molecule. The shortest metal–metal separation is 4.6038 (11) Å. The molecular structure is stabilized by intramolecular C—H···O hydrogen-bonding interactions.

Related literature

For general background, see: Belicchi Ferrari *et al.* (2000); Chatterjee & Mitra (1999); Chatterjee *et al.* (2000); Kureshy *et al.* (2000); Shi *et al.* (2001). For related structures, see: Bermejo *et al.* (1999); Butcher *et al.* (1981); Yang *et al.* (2003); Yang & Lin (2005).



Experimental

Crystal data

$[Ni_3(C_{15}H_{12}N_2O_4)_2(C_5H_5N)_2(C_3H_6NO)_2]$
M_r = 1046.98
Monoclinic, $C2/c$
a = 28.646 (6) Å
b = 8.2490 (16) Å
c = 21.176 (4) Å
β = 112.98 (3)°
V = 4606.8 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.28 mm^{-1}
T = 298 (2) K
0.21 × 0.18 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: none
21350 measured reflections
5266 independent reflections
3189 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.129
S = 1.00
5266 reflections
306 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2	0.93	2.41	2.749 (5)	101
C16—H16···O3	0.93	2.31	2.800 (6)	113
C20—H20···O1	0.93	2.28	2.783 (8)	114
C23—H23C···O5	0.96	2.34	2.752 (9)	105
C9—H9B···O2^{i}	0.97	2.38	3.183 (6)	139
C21—H21A···O2^{i}	0.93	2.40	2.968 (8)	119

Symmetry code: (i) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

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: *SHELXTL/PC* (Sheldrick, 1993); 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: RZ2145).

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supplementary materials

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Bis(dimethylformamide-2κO)bis[μ-1-(2-oxidobenzoyl)-2-(phenoxyacetyl)hydrazine(3-)]-1$\kappa^3 O,N,O'$:2$\kappa^2 N,O''$;2$\kappa^2 N,O''$:3$\kappa^3 O,N,O'$-dipyridine-1κN,3κN-trinickel(II)

Y. Zhao, J.-H. Luo, X.-H. Huang, C.-C. Huang and D.-S. Liu

Comment

Hydrazide and its analogues have found practical applications in catalysis (Chatterjee & Mitra, 1999; Chatterjee *et al.*, 2000; Kureshy *et al.*, 2000) and in biochemistry (Belicchi Ferrari *et al.*, 2000; Shi *et al.*, 2001). *N*-Acylsalicylhydrazides, which contain several donor atoms, are strong chelating agents and are particularly well suited for the preparation of multinuclear complexes. We report here the synthesis and crystal structure of a new trinuclear nickel(II) complex, (I), containing the *N*-phenoxyacetylsalicylhydrazidate ligand.

The trinuclear nickel(II) complex possesses a crystallographycally imposed centre of symmetry. In the molecule, the central nickel atom Ni2 (Fig.1) has an axially elongated octahedral coordination of Ni(ON)(ON)(O)(O) type. The Ni2—O2 and Ni2—N2 bond distances at the equatorial plane (2.007 (3) and 2.047 (3) Å respectively) are shorter than the corresponding bond lengths in similar nickel(II) complexes (Yang *et al.*, 2003; Yang & Lin, 2005; Butcher *et al.*, 1981; Bermejo *et al.*, 1999). The O atoms of the two dimethylformamide molecules occupy the axial positions at longer distances [Ni2—O5 = 2.147 (3) Å]. The outer Ni atoms (Ni1, Ni1A) adopt a square-planar geometry. The Ni—N(hydrazide) and Ni—N(py) bond distances are 1.838 (4) and 1.940 (1) Å, respectively. The Ni—*O*(carbonyl) distances are 1.814 (36) and 1.854 (38) Å, respectively. The Ni1···Ni2 distance is 4.6038 (11) Å, whereas the Ni1···Ni1A separation is 9.208 (2) Å, which is in good agreement with the corresponding values [9.2030 (8) − 9.1876 (9) Å] reported for bis[μ-(*N*-butylsalicylhydrazidate)(pyridine)nickel(II)]bispyridinenickel(II) (Yang *et al.*, 2003) and shorter than that found in bis(μ_2-*N*'-benzoyl-2-oxybenzoylhydrazidato)-bis(dimethylformamide)- dipyridine-trinickel(ii) [9.3038 (7) Å; Yang & Lin, 2005). The molecular structure is stabilized by intramolecular C—H···O hydrogen bonds (Table 1).

Experimental

Two drops of pyridine were added to a solution of *N*-phenoxyacetylsalicylhydrazide (0.0286 g, 0.1 mmol) and $NiCl_2{\cdot}6H_2O$ (0.0475 g, 0.2 mmol) in ethanol (3 ml), chloroform (7 ml) and DMF (2 ml). The resulting red solution was stirred for 2 h and then filtered. Red crystals suitable for X-ray analysis were obtained after 6 days on slow evaporation of the solvent.

Refinement

All H atoms were placed in idealized positions an refined using the riding-model approximation, with C—H = 0.93–0.97 Å, and with U_{iso} (H) = 1.2U_{eq}(C) or 1.5 U_{eq}(C) for the methyl groups.

Figures

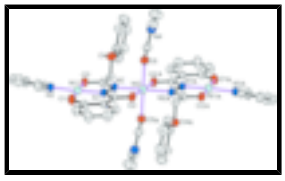


Fig. 1. View of the title compound with 30% probability displacement ellipsoids. H atoms have been omitted for clarity. [symmetry code: (A) 1/2 − *x*, 3/2 − *y*, 1 − *z*]

Bis(dimethylformamide-2κ*O*)bis[μ-1-(2-oxidobenzoyl)-2- (phenoxyacetyl)hydrazine(3-)]-1κ^3*O*,*N*,*O*':2κ^2*N*,*O*'';2κ^2*N*,*O*'':3κ^3*O*,*N*,*O*'-dipyridine-1κ*N*,3κ*N*- trinickel(II)

Crystal data

$[Ni_3(C_{15}H_{12}N_2O_4)_2(C_5H_5N)_2(C_3H_6NO)_2]$	F_{000} = 2168
M_r = 1046.98	D_x = 1.510 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 13455 reflections
a = 28.646 (6) Å	θ = 3.1–27.5º
b = 8.2490 (16) Å	μ = 1.28 mm^{-1}
c = 21.176 (4) Å	*T* = 298 (2) K
β = 112.98 (3)º	Block, red
V = 4606.8 (19) Å^3	0.21 × 0.18 × 0.18 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer	3189 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.065
Monochromator: graphite	θ_{max} = 27.5º
T = 298(2) K	θ_{min} = 3.1º
ω scans	*h* = −37→35
Absorption correction: none	*k* = −10→10
21350 measured reflections	*l* = −27→27
5266 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.060	H-atom parameters constrained
$wR(F^2)$ = 0.129	$w = 1/[\sigma^2(F_o^2) + (0.008P)^2 + 4.5359P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max} < 0.001$

5266 reflections	$\Delta\rho_{max} = 0.50$ e Å^{-3}
306 parameters	$\Delta\rho_{min} = -0.64$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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}
Ni1	0.19205 (3)	0.36882 (8)	0.32794 (3)	0.0538 (2)
Ni2	0.2500	0.7500	0.5000	0.0429 (2)
O1	0.13443 (13)	0.4435 (4)	0.26024 (17)	0.0628 (10)
O2	0.18921 (12)	0.8037 (4)	0.41488 (14)	0.0496 (8)
O3	0.25040 (12)	0.2981 (4)	0.39945 (15)	0.0520 (8)
O4	0.35544 (13)	0.3998 (4)	0.49849 (16)	0.0593 (9)
O5	0.29860 (14)	0.8574 (4)	0.45592 (17)	0.0581 (9)
N1	0.20072 (14)	0.5505 (4)	0.38186 (18)	0.0451 (9)
N2	0.24345 (14)	0.5421 (4)	0.44438 (17)	0.0443 (9)
N3	0.18937 (17)	0.1722 (5)	0.2764 (2)	0.0567 (11)
N4	0.37718 (18)	0.9207 (5)	0.4632 (2)	0.0657 (12)
C1	0.11199 (19)	0.5850 (6)	0.2586 (2)	0.0547 (13)
C2	0.12931 (17)	0.7048 (6)	0.3092 (2)	0.0480 (11)
C3	0.10198 (19)	0.8496 (7)	0.3000 (3)	0.0610 (14)
H3	0.1135	0.9294	0.3336	0.073*
C4	0.0593 (2)	0.8768 (8)	0.2436 (3)	0.0835 (19)
H4	0.0420	0.9746	0.2385	0.100*
C5	0.0417 (2)	0.7597 (8)	0.1943 (3)	0.087 (2)
H5	0.0120	0.7767	0.1559	0.105*
C6	0.0679 (2)	0.6173 (8)	0.2014 (3)	0.0718 (16)
H6	0.0558	0.5398	0.1669	0.086*
C7	0.17486 (18)	0.6884 (5)	0.3720 (2)	0.0447 (11)
C8	0.26588 (18)	0.4049 (5)	0.4479 (2)	0.0439 (11)
C9	0.31135 (18)	0.3611 (6)	0.5110 (2)	0.0493 (11)
H9B	0.3112	0.4217	0.5502	0.059*
H9A	0.3109	0.2463	0.5208	0.059*
C10	0.40053 (19)	0.3535 (6)	0.5476 (3)	0.0546 (12)
C11	0.4439 (2)	0.4055 (8)	0.5393 (3)	0.0760 (17)

H11	0.4409	0.4689	0.5016	0.091*
C12	0.4915 (3)	0.3640 (9)	0.5867 (4)	0.094 (2)
H12	0.5201	0.4019	0.5808	0.113*
C13	0.4971 (3)	0.2704 (9)	0.6407 (4)	0.091 (2)
H13	0.5293	0.2421	0.6719	0.109*
C14	0.4558 (3)	0.2182 (8)	0.6493 (3)	0.0812 (18)
H14	0.4598	0.1530	0.6869	0.097*
C15	0.4065 (2)	0.2583 (7)	0.6035 (3)	0.0648 (14)
H15	0.3785	0.2210	0.6109	0.078*
C16	0.2155 (2)	0.0391 (6)	0.3074 (3)	0.0657 (15)
H16	0.2327	0.0396	0.3548	0.079*
C17	0.2178 (3)	−0.0952 (7)	0.2721 (3)	0.0758 (17)
H17	0.2363	−0.1850	0.2951	0.091*
C18	0.1931 (3)	−0.0984 (8)	0.2031 (4)	0.087 (2)
H18	0.1942	−0.1902	0.1782	0.105*
C19	0.1673 (3)	0.0321 (9)	0.1715 (3)	0.095 (2)
H19	0.1512	0.0334	0.1239	0.114*
C20	0.1642 (3)	0.1681 (8)	0.2097 (3)	0.086 (2)
H20	0.1442	0.2560	0.1875	0.104*
C21	0.3443 (2)	0.8379 (7)	0.4809 (3)	0.0626 (14)
H21A	0.3571	0.7595	0.5149	0.075*
C22	0.4316 (3)	0.8982 (10)	0.4983 (4)	0.109 (3)
H22A	0.4383	0.8200	0.5343	0.163*
H22B	0.4472	0.9996	0.5175	0.163*
H22C	0.4453	0.8602	0.4662	0.163*
C23	0.3596 (3)	1.0451 (7)	0.4122 (3)	0.0814 (18)
H23A	0.3644	1.0111	0.3717	0.122*
H23B	0.3785	1.1428	0.4296	0.122*
H23C	0.3243	1.0650	0.4011	0.122*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0654 (4)	0.0466 (4)	0.0479 (4)	−0.0014 (3)	0.0205 (3)	−0.0051 (3)
Ni2	0.0505 (5)	0.0389 (4)	0.0367 (4)	0.0024 (4)	0.0142 (4)	−0.0023 (4)
O1	0.062 (2)	0.058 (2)	0.053 (2)	0.0038 (19)	0.0064 (17)	−0.0095 (17)
O2	0.062 (2)	0.0417 (17)	0.0393 (16)	0.0069 (16)	0.0136 (15)	−0.0028 (14)
O3	0.064 (2)	0.0437 (17)	0.0460 (17)	0.0045 (16)	0.0189 (16)	−0.0048 (15)
O4	0.059 (2)	0.067 (2)	0.0518 (19)	0.0059 (19)	0.0218 (17)	0.0145 (18)
O5	0.066 (2)	0.057 (2)	0.054 (2)	−0.0014 (19)	0.0260 (18)	0.0030 (17)
N1	0.048 (2)	0.044 (2)	0.0387 (19)	0.0011 (18)	0.0132 (17)	−0.0026 (17)
N2	0.056 (2)	0.039 (2)	0.0330 (18)	0.0046 (19)	0.0130 (17)	−0.0024 (16)
N3	0.072 (3)	0.051 (2)	0.052 (2)	−0.008 (2)	0.030 (2)	−0.008 (2)
N4	0.062 (3)	0.059 (3)	0.084 (3)	0.001 (2)	0.037 (3)	0.003 (2)
C1	0.055 (3)	0.055 (3)	0.049 (3)	−0.005 (3)	0.014 (2)	−0.002 (2)
C2	0.043 (3)	0.053 (3)	0.045 (2)	0.000 (2)	0.014 (2)	0.002 (2)
C3	0.052 (3)	0.062 (3)	0.059 (3)	0.006 (3)	0.011 (3)	−0.007 (3)
C4	0.068 (4)	0.075 (4)	0.084 (4)	0.024 (3)	0.004 (3)	0.001 (4)

C5	0.065 (4)	0.086 (5)	0.076 (4)	0.008 (4)	−0.011 (3)	−0.012 (4)
C6	0.058 (3)	0.077 (4)	0.061 (3)	−0.006 (3)	0.002 (3)	−0.012 (3)
C7	0.054 (3)	0.043 (2)	0.041 (2)	0.004 (2)	0.022 (2)	0.002 (2)
C8	0.053 (3)	0.042 (2)	0.040 (2)	0.000 (2)	0.021 (2)	0.003 (2)
C9	0.063 (3)	0.045 (3)	0.042 (2)	0.008 (2)	0.023 (2)	0.005 (2)
C10	0.055 (3)	0.053 (3)	0.058 (3)	0.006 (3)	0.023 (3)	0.000 (3)
C11	0.062 (4)	0.080 (4)	0.090 (4)	−0.002 (3)	0.035 (3)	0.013 (4)
C12	0.061 (4)	0.092 (5)	0.127 (6)	−0.004 (4)	0.034 (4)	−0.007 (5)
C13	0.069 (4)	0.080 (5)	0.103 (5)	0.011 (4)	0.011 (4)	−0.010 (4)
C14	0.074 (4)	0.083 (4)	0.073 (4)	0.014 (4)	0.013 (3)	0.009 (3)
C15	0.055 (3)	0.075 (4)	0.061 (3)	0.014 (3)	0.020 (3)	0.009 (3)
C16	0.085 (4)	0.051 (3)	0.066 (3)	−0.003 (3)	0.035 (3)	−0.004 (3)
C17	0.102 (5)	0.058 (3)	0.082 (4)	−0.004 (3)	0.051 (4)	−0.016 (3)
C18	0.111 (6)	0.071 (4)	0.096 (5)	−0.017 (4)	0.059 (4)	−0.034 (4)
C19	0.125 (6)	0.084 (5)	0.072 (4)	−0.003 (5)	0.033 (4)	−0.019 (4)
C20	0.122 (6)	0.073 (4)	0.056 (3)	−0.023 (4)	0.026 (4)	−0.021 (3)
C21	0.075 (4)	0.055 (3)	0.063 (3)	0.010 (3)	0.032 (3)	0.005 (3)
C22	0.078 (5)	0.107 (6)	0.147 (7)	0.007 (4)	0.051 (5)	0.019 (5)
C23	0.105 (5)	0.066 (4)	0.088 (4)	−0.007 (4)	0.054 (4)	0.007 (3)

Geometric parameters (Å, °)

Ni1—O1	1.820 (3)	C5—H5	0.9300
Ni1—N1	1.841 (4)	C6—H6	0.9300
Ni1—O3	1.858 (3)	C8—C9	1.502 (6)
Ni1—N3	1.939 (4)	C9—H9B	0.9700
Ni2—O2^{i}	2.007 (3)	C9—H9A	0.9700
Ni2—O2	2.007 (3)	C10—C15	1.374 (7)
Ni2—N2	2.047 (3)	C10—C11	1.388 (7)
Ni2—N2^{i}	2.047 (3)	C11—C12	1.384 (8)
Ni2—O5	2.147 (4)	C11—H11	0.9300
Ni2—O5^{i}	2.147 (4)	C12—C13	1.337 (9)
O1—C1	1.326 (6)	C12—H12	0.9300
O2—C7	1.267 (5)	C13—C14	1.336 (9)
O3—C8	1.292 (5)	C13—H13	0.9300
O4—C10	1.359 (6)	C14—C15	1.405 (7)
O4—C9	1.425 (6)	C14—H14	0.9300
O5—C21	1.215 (6)	C15—H15	0.9300
N1—C7	1.329 (5)	C16—C17	1.353 (7)
N1—N2	1.411 (5)	C16—H16	0.9300
N2—C8	1.289 (5)	C17—C18	1.352 (8)
N3—C20	1.313 (6)	C17—H17	0.9300
N3—C16	1.346 (6)	C18—C19	1.330 (9)
N4—C21	1.332 (7)	C18—H18	0.9300
N4—C23	1.431 (7)	C19—C20	1.406 (8)
N4—C22	1.453 (7)	C19—H19	0.9300
C1—C6	1.392 (7)	C20—H20	0.9300
C1—C2	1.399 (6)	C21—H21A	0.9300

C2—C3	1.399 (7)	C22—H22A	0.9600
C2—C7	1.462 (6)	C22—H22B	0.9600
C3—C4	1.354 (7)	C22—H22C	0.9600
C3—H3	0.9300	C23—H23A	0.9600
C4—C5	1.366 (8)	C23—H23B	0.9600
C4—H4	0.9300	C23—H23C	0.9600
C5—C6	1.370 (8)		
O1—Ni1—N1	94.27 (16)	N1—C7—C2	117.8 (4)
O1—Ni1—O3	177.75 (15)	N2—C8—O3	123.0 (4)
N1—Ni1—O3	83.66 (15)	N2—C8—C9	120.1 (4)
O1—Ni1—N3	90.88 (17)	O3—C8—C9	116.9 (4)
N1—Ni1—N3	174.67 (18)	O4—C9—C8	107.7 (4)
O3—Ni1—N3	91.21 (16)	O4—C9—H9B	110.2
$O2^{i}$—Ni2—O2	180.00 (17)	C8—C9—H9B	110.2
$O2^{i}$—Ni2—N2	100.61 (13)	O4—C9—H9A	110.2
O2—Ni2—N2	79.39 (13)	C8—C9—H9A	110.2
$O2^{i}$—Ni2—$N2^{i}$	79.39 (13)	H9B—C9—H9A	108.5
O2—Ni2—$N2^{i}$	100.61 (13)	O4—C10—C15	125.6 (5)
N2—Ni2—$N2^{i}$	180.000 (1)	O4—C10—C11	116.5 (5)
$O2^{i}$—Ni2—O5	89.95 (14)	C15—C10—C11	117.9 (5)
O2—Ni2—O5	90.05 (14)	C12—C11—C10	120.6 (6)
N2—Ni2—O5	91.43 (14)	C12—C11—H11	119.7
$N2^{i}$—Ni2—O5	88.57 (14)	C10—C11—H11	119.7
$O2^{i}$—Ni2—$O5^{i}$	90.05 (14)	C13—C12—C11	121.3 (7)
O2—Ni2—$O5^{i}$	89.95 (14)	C13—C12—H12	119.4
N2—Ni2—$O5^{i}$	88.57 (14)	C11—C12—H12	119.4
$N2^{i}$—Ni2—$O5^{i}$	91.43 (14)	C14—C13—C12	119.0 (7)
O5—Ni2—$O5^{i}$	180.00 (13)	C14—C13—H13	120.5
C1—O1—Ni1	126.9 (3)	C12—C13—H13	120.5
C7—O2—Ni2	113.7 (3)	C13—C14—C15	122.3 (6)
C8—O3—Ni1	110.7 (3)	C13—C14—H14	118.9
C10—O4—C9	116.2 (4)	C15—C14—H14	118.9
C21—O5—Ni2	121.8 (4)	C10—C15—C14	118.9 (6)
C7—N1—N2	114.5 (4)	C10—C15—H15	120.5
C7—N1—Ni1	131.9 (3)	C14—C15—H15	120.5
N2—N1—Ni1	113.6 (3)	N3—C16—C17	122.4 (6)
C8—N2—N1	109.1 (3)	N3—C16—H16	118.8
C8—N2—Ni2	140.4 (3)	C17—C16—H16	118.8
N1—N2—Ni2	110.5 (3)	C18—C17—C16	119.7 (6)
C20—N3—C16	118.1 (5)	C18—C17—H17	120.1
C20—N3—Ni1	120.8 (4)	C16—C17—H17	120.1
C16—N3—Ni1	121.0 (3)	C19—C18—C17	118.8 (6)
C21—N4—C23	120.1 (5)	C19—C18—H18	120.6
C21—N4—C22	121.8 (5)	C17—C18—H18	120.6
C23—N4—C22	117.9 (5)	C18—C19—C20	120.3 (6)
O1—C1—C6	117.2 (5)	C18—C19—H19	119.9

O1—C1—C2	125.3 (4)	C20—C19—H19	119.9
C6—C1—C2	117.4 (5)	N3—C20—C19	120.6 (7)
C1—C2—C3	119.0 (4)	N3—C20—H20	119.7
C1—C2—C7	123.7 (4)	C19—C20—H20	119.7
C3—C2—C7	117.2 (4)	O5—C21—N4	124.7 (5)
C4—C3—C2	121.9 (5)	O5—C21—H21A	117.6
C4—C3—H3	119.1	N4—C21—H21A	117.6
C2—C3—H3	119.1	N4—C22—H22A	109.5
C3—C4—C5	119.6 (6)	N4—C22—H22B	109.5
C3—C4—H4	120.2	H22A—C22—H22B	109.5
C5—C4—H4	120.2	N4—C22—H22C	109.5
C4—C5—C6	120.0 (5)	H22A—C22—H22C	109.5
C4—C5—H5	120.0	H22B—C22—H22C	109.5
C6—C5—H5	120.0	N4—C23—H23A	109.5
C5—C6—C1	122.2 (5)	N4—C23—H23B	109.5
C5—C6—H6	118.9	H23A—C23—H23B	109.5
C1—C6—H6	118.9	N4—C23—H23C	109.5
O2—C7—N1	121.7 (4)	H23A—C23—H23C	109.5
O2—C7—C2	120.5 (4)	H23B—C23—H23C	109.5

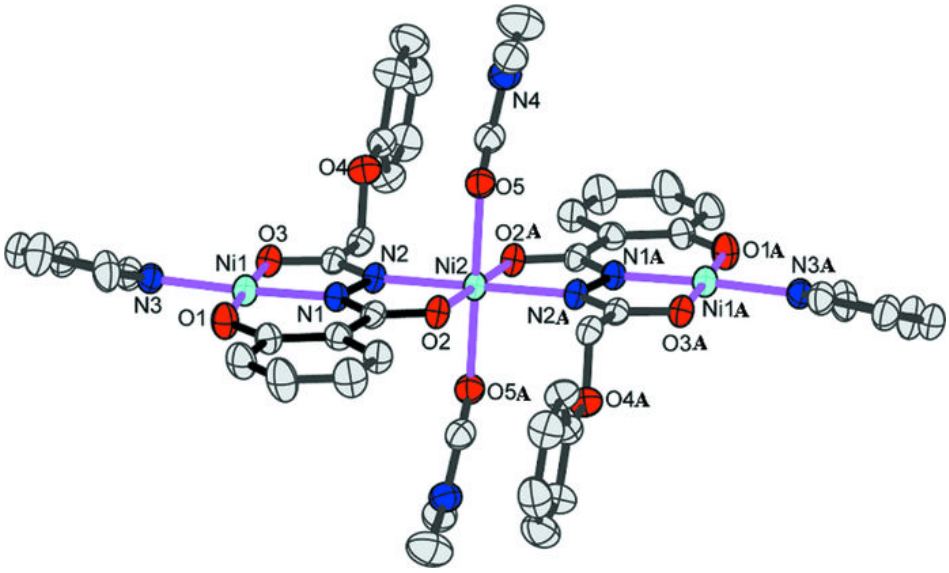
Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2	0.93	2.41	2.749 (5)	101
C16—H16···O3	0.93	2.31	2.800 (6)	113
C20—H20···O1	0.93	2.28	2.783 (8)	114
C23—H23C···O5	0.96	2.34	2.752 (9)	105
C9—H9B···O2^{i}	0.97	2.38	3.183 (6)	139
C21—H21A···O2^{i}	0.93	2.40	2.968 (8)	119

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

Fig. 1


N4
O4
O5
O3
Ni1
N2
Ni2
O2A
N1A
O1A
N3A
N3
O1
N1
O2
N2A
Ni1A
O3A
O5A
O4A