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Crystal structure of bis{4-[(4-methylbenzyl)oxy]-*N*′-(4-methylbenzylidene)benzohydrazidato}nickel(II)

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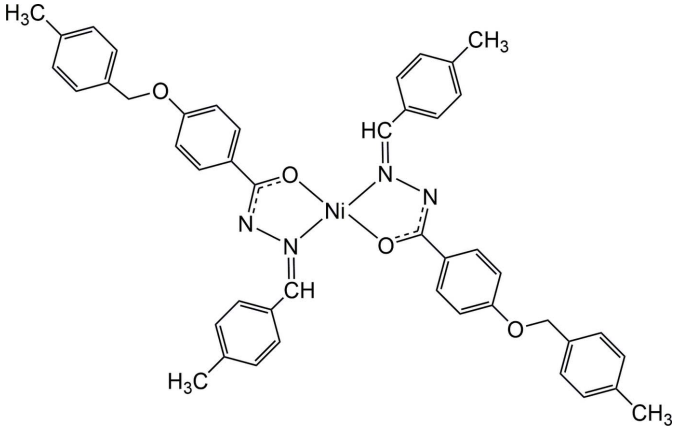
In the title complex, $[Ni(C_{23}H_{21}N_2O_2)_2]$, the central Ni^{II} atom is located on an inversion centre and exhibits a slightly distorted square-planar N_2O_2 coordination environment. A *trans*-configuration of the *N*,*O* chelating ligands results from the imposed site symmetry of the central Ni^{II} atom. In the crystal, individual molecules stack along the *a* axis through weak π–π stacking interactions between the phenyl rings.

1. Chemical context

Variously substituted hydrazone ligands have attracted special attention because of their chelating capabilities and structural properties, such as the degree of rigidity, a conjugated π-system and an N—H unit that readily participates in hydrogen bonding and may be easily deprotonated. The corresponding nickel(II) complexes are of considerable interest since they exhibit a broad spectrum of physiological and pharmacological activities (Yang *et al.*, 2020; Al-Qadsy *et al.*, 2021; Neethu *et al.*, 2021; Krishnamoorthy *et al.*, 2012), most of which are structure-dependent properties.



We report here the synthesis and crystal structure of another Ni^{II} complex with a derivatized hydrazone ligand.

2. Structural commentary

The central metal Ni^{II} atom of the title complex is located on an inversion center. Hence, the asymmetric unit comprises half a molecule (Fig. 1). The enolizable O atom and the

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O1^{i}	0.95	2.38	2.9455 (18)	118
C3—H3···N2	0.95	2.37	2.945 (2)	118
C11—H11···O1	0.95	2.43	2.7590 (19)	100

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

azomethine N atom of the ligand coordinate to the Ni^{II} atom to form a five-membered chelate ring. The Ni^{II} atom exhibits a slightly distorted square-planar coordination environment with the deprotonated ligands in a *trans* configuration imposed by the crystal symmetry. The Ni—N1 and Ni—O1 bond lengths are 1.8677 (12) and 1.8363 (10) Å, respectively, with a chelating angle of 83.47 (5)°. These data are in agreement with previously reported crystal structures of related complexes (Yang *et al.*, 2020; Al-Qadsy *et al.*, 2021; Neethu *et al.*, 2021; Krishnamoorthy *et al.*, 2012), irrespective of the substituents present in the ligand.

As expected, the C9—O1 bond length of 1.3009 (18) Å lies between a C—O single bond (1.43 Å; Allen *et al.*, 1987) and a C=O double bond (1.21 Å; Allen *et al.*, 1987). The bond lengths N1—C8 of 1.2977 (19) Å and N2—C9 of 1.3145 (18) Å are close to the value of a typical C=N bond (1.30 Å; Allen *et al.*, 1987). These data reveal that the —CH=N—N=C—O fragment of the ligand remains a conjugated system even after the loss of a H atom from its enolized carbonyl O atom. The complex is stabilized by weak intramolecular C8—H8···O1, C3—H3···N2 and C11—H11···O1 hydrogen bonds involving phenyl and methylene donor groups and the coordinating atoms as acceptor groups (Table 1). The benzylidene ring is tilted by 26.06 (6)° with respect to the N_2O_2 coordination plane, while the phenyl rings of the ether moiety form a dihedral angle of 83.29 (5)°.

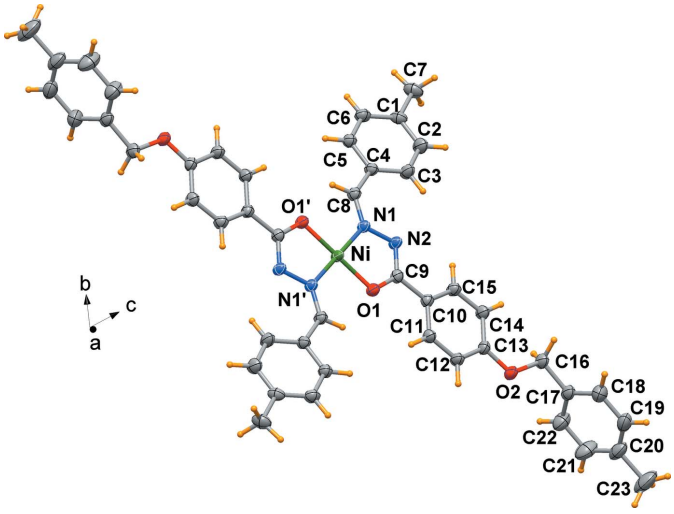


Figure 1
Molecular structure of the centrosymmetric nickel(II) complex, drawn with displacement ellipsoids at the 50% probability level. [symmetry code for primed atoms: $-x+2, -y, -z+2$.]

The bond-valence sum (BVS) calculated for the Ni^{II} atom present in the complex, using the parameters of Brese & O'Keeffe (1991), indicate a higher value (2.97 valence units) than expected for a formal ionic charge of +2. The calculated high value can be reasonably attributed to a very pronounced covalent bonding associated with the Ni—O and Ni—N bonds. As a matter of fact, a set of new optimized r_0 parameters to be used for the BVS calculation for model compounds involving Ni^{II}—O, Ni^{II}—S, Ni^{II}—N interactions has been proposed (Liu & Thorp, 1993). By using these values, the BVS calculation for this complex gives a value of 2.36 valence units.

3. Supramolecular features

Individual molecular complexes are weakly packed along the *a* axis through π-ring interactions involving the phenyl rings, with centroid-to-centroid distances of 4.6914 (2) Å and a slippage of *ca* 3.0-3.3 Å, as shown in Fig. 2. In addition, the five-membered chelate rings of neighbouring complexes have even shorter distances [3.4555 (2) Å with a slippage of 0.96 Å].

4. Database survey

A search in the Cambridge Crystal Structure Database (CSD, version 5.43, update June 2022); Groom *et al.*, 2016) retrieved more than twenty bis-chelated square-planar nickel(II) complexes with hydrazone-based ligands also bearing bulky ferrocenyl groups (Krishnamoorthy *et al.*, 2012), 2,2′-bithiophenyl (Yang *et al.*, 2020) or 9-anthrylmethylene fragments (Mondal *et al.*, 2014). However, no species comprising a long benzyl-phenyl ether chain has been reported so far. It is worth noting that all characterized Ni^{II} complexes exhibit a *trans*-configuration of ligands, where the —CH=N—N=C—O fragment is chelating, and the coordination Ni—O and Ni—N bond lengths do not appear to be significantly affected by the electronic or steric properties of groups present on the ligands.

5. Synthesis and crystallization

To a solution of 4-(4-methylbenzyloxy)benzoylhydrazine (0.26 g, 1 mmol in 25 ml of ethanol), 4-methyl benzaldehyde

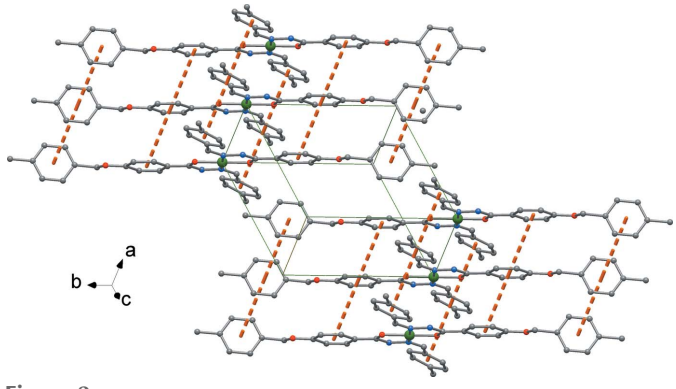


Figure 2
Crystal packing of individual complexes showing the π-ring interactions as dotted lines.

(0.12 g, 1 mmol) was added and the mixture was refluxed for half an hour. A solution of nickel(II) acetate tetrahydrate (0.13 g, 0.5 mmol in 5 ml of ethanol) was then added and refluxing was continued for 2 h. The obtained orange precipitate was filtered off and washed three times with hot ethanol. The product was recrystallized from a mixture of chloroform and acetonitrile (5:1, *v*/*v*) and orange crystals, suitable for X-ray diffraction, were filtered off, washed with hot ethanol, and left to dry in a desiccator over silica gel. Yield: 0.45 g, 58%. Melting point: >523 K. FT–IR: 1603, 1585 ν (C═N—N═C), 486 ν (*M*—N), 503 ν (*M*—O). LC–MS (ESI) *m*/*z*: $[M + H]^+$. Calculated for $C_{46}H_{42}N_4O_4Ni$ 773.2632; found 773.2636. μ_{eff}: 0.832 B·M. Molar conductance (ohm^{-1} cm^2 mol^{-1}): 1.0. NMR spectra were not obtained due to the low solubility of the complex even in DMSO.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydrogen atoms were included in idealized positions as riding contributions with fixed isotropic displacement parameters [C—H = 0.95–0.99 Å; U_{iso}(H) = 1.2 or 1.5 U_{eq}(C)].

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Funding information

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Table 2
Experimental details.

Crystal data	
Chemical formula	$[Ni(C_{23}H_{21}N_2O_2)_2]$
M_r	773.54
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	173
a, *b*, *c* (Å)	4.6914 (2), 13.0677 (7), 16.9923 (8)
α, β, γ (°)	68.441 (5), 83.739 (6), 88.032 (6)
V (Å^3)	963.05 (9)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.55
Crystal size (mm)	0.32 × 0.08 × 0.03
Data collection	
Diffractometer	Rigaku R-AXIS RAPID
Absorption correction	Multi-scan (*ABSCOR*; Rigaku, 1995)
T_{min}, T_{max}	0.761, 0.984
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9456, 4375, 3883
R_{int}	0.024
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.037, 0.096, 1.06
No. of reflections	4375
No. of parameters	252
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.42, −0.19

Computer programs: *CrystalStructure* (Rigaku, 2018), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg, 1999) and *WinGX* (Farrugia, 2012).

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Robin Taylor, R. (1987). *J. Chem. Soc., Perkin Trans. 2*, pp. S1–S19.

Al-Qadsy, I., Al-Odayni, A.-B., Saeed, W. S., Alrabie, A., Al-Adhreai, A., Al-Faqeeh, L. A. S., Lama, P., Alghamdi, A. A. & Farooqui, M. (2021). *Crystals*, **11**, 110.

Brandenburg, K. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Brese, N. E. & O'Keeffe, M. (1991). *Acta Cryst.* B**47**, 192–197.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Krishnamoorthy, P., Sathyadevi, P., Butorac, R. R., Cowley, A. H., Bhuvanesh, N. S. P. & Dharmaraj, N. (2012). *Dalton Trans.* **41**, 4423–4436.

Liu, W. & Thorp, H. H. (1993). *Inorg. Chem.* **32**, 4102–4105.

Mondal, S., Das, C., Ghosh, B., Pakhira, B., Blake, A. J., Drew, M. G. B. & Chattopadhyay, S. K. (2014). *Polyhedron*, **80**, 272–281.

Neethu, K. S., Sivaselvam, S., Theetharappan, M., Ranjitha, J., Bhuvanesh, N. S. P., Ponpandian, N., Neelakantan, M. A. & Kaveri, M. V. (2021). *Inorg. Chim. Acta*, **524**, 120419.

Rigaku (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.

Rigaku (2018). *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Yang, P., Chen, H., Wang, Z.-Z., Zhang, L.-L., Zhang, D.-D., Shi, Q.-S. & Xie, X.-B. (2020). *J. Inorg. Biochem.* **213**, 111248.

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Computing details

Data collection: *CrystalStructure* (Rigaku, 2018); cell refinement: *CrystalStructure* (Rigaku, 2018); data reduction: *CrystalStructure* (Rigaku, 2018); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Bis{4-[(4-methylbenzyl)oxy]-*N*′-(4-methylbenzylidene)benzohydrazidato}nickel(II)

Crystal data

$[Ni(C_{23}H_{21}N_2O_2)_2]$
M_r = 773.54
Triclinic, $P\overline{1}$
a = 4.6914 (2) Å
b = 13.0677 (7) Å
c = 16.9923 (8) Å
α = 68.441 (5)°
β = 83.739 (6)°
γ = 88.032 (6)°
V = 963.05 (9) Å^3
Z = 1
$F(000)$ = 406
D_x = 1.334 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 8457 reflections
θ = 1.7–27.5°
μ = 0.55 mm^{-1}
T = 173 K
Platelet, orange
0.32 × 0.08 × 0.03 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (ABSCOR; Rigaku, 1995)
T_{min} = 0.761, T_{max} = 0.984
9456 measured reflections
4375 independent reflections
3883 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.5°, θ_{min} = 2.5°
h = −6→5
k = −16→16
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.096
S = 1.06
4375 reflections
252 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 0.1727P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	1.000000	0.000000	1.000000	0.02390 (10)
O1	0.8708 (2)	0.13421 (8)	0.93185 (7)	0.0267 (2)
O2	0.2680 (3)	0.52033 (9)	0.65888 (7)	0.0339 (3)
N1	0.7701 (3)	−0.05385 (10)	0.94061 (8)	0.0250 (3)
N2	0.6329 (3)	0.02767 (10)	0.87729 (8)	0.0272 (3)
C1	0.1840 (3)	−0.32649 (13)	0.85447 (11)	0.0313 (3)
C2	0.2533 (4)	−0.21651 (14)	0.80907 (11)	0.0389 (4)
H2	0.183365	−0.181019	0.755246	0.047*
C3	0.4201 (4)	−0.15681 (13)	0.83933 (11)	0.0354 (4)
H3	0.457644	−0.081018	0.807386	0.042*
C4	0.5340 (3)	−0.20830 (12)	0.91727 (10)	0.0267 (3)
C5	0.4694 (4)	−0.31917 (12)	0.96220 (10)	0.0302 (3)
H5	0.546693	−0.355980	1.014716	0.036*
C6	0.2951 (4)	−0.37703 (13)	0.93214 (11)	0.0325 (3)
H6	0.250866	−0.452077	0.964938	0.039*
C7	−0.0032 (4)	−0.38838 (15)	0.82047 (13)	0.0408 (4)
H7A	−0.191485	−0.353241	0.813325	0.049*
H7B	−0.026408	−0.464591	0.860552	0.049*
H7C	0.086306	−0.387620	0.765383	0.049*
C8	0.7190 (3)	−0.15674 (12)	0.95597 (10)	0.0269 (3)
H8	0.818971	−0.207025	0.999831	0.032*
C9	0.7016 (3)	0.12306 (12)	0.87971 (9)	0.0248 (3)
C10	0.5783 (3)	0.22452 (12)	0.82102 (9)	0.0246 (3)
C11	0.6537 (4)	0.32665 (12)	0.82264 (10)	0.0285 (3)
H11	0.782209	0.329648	0.861251	0.034*
C12	0.5427 (4)	0.42268 (12)	0.76866 (10)	0.0311 (3)
H12	0.592488	0.491322	0.770952	0.037*
C13	0.3584 (3)	0.41942 (12)	0.71090 (9)	0.0272 (3)
C14	0.2802 (4)	0.31891 (13)	0.70838 (10)	0.0300 (3)
H14	0.154229	0.316308	0.669017	0.036*
C15	0.3889 (4)	0.22253 (12)	0.76416 (10)	0.0289 (3)
H15	0.332585	0.153781	0.763416	0.035*
C16	0.1042 (4)	0.52370 (13)	0.59118 (10)	0.0338 (4)
H16A	−0.077691	0.482264	0.614859	0.041*
H16B	0.214098	0.489949	0.553892	0.041*
C17	0.0433 (4)	0.64249 (13)	0.54117 (10)	0.0342 (4)
C18	0.1936 (5)	0.69725 (15)	0.46333 (12)	0.0467 (5)
H18	0.341540	0.660436	0.441520	0.056*
C19	0.1304 (6)	0.80648 (16)	0.41616 (13)	0.0533 (5)

H19	0.235121	0.842724	0.362308	0.064*
C20	−0.0782 (5)	0.86203 (16)	0.44579 (14)	0.0549 (6)
C21	−0.2279 (6)	0.80699 (18)	0.52500 (17)	0.0641 (6)
H21	−0.372618	0.844499	0.547317	0.077*
C22	−0.1685 (5)	0.69813 (16)	0.57170 (14)	0.0490 (5)
H22	−0.274742	0.661553	0.625238	0.059*
C23	−0.1500 (7)	0.98005 (19)	0.3935 (2)	0.0871 (10)
H23A	−0.100061	1.028406	0.422521	0.104*
H23B	−0.355632	0.985730	0.387091	0.104*
H23C	−0.040649	1.002234	0.337299	0.104*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02565 (16)	0.01922 (14)	0.02805 (16)	0.00079 (10)	−0.01098 (11)	−0.00788 (11)
O1	0.0289 (6)	0.0218 (5)	0.0305 (5)	0.0003 (4)	−0.0119 (4)	−0.0083 (4)
O2	0.0470 (7)	0.0234 (5)	0.0318 (6)	0.0040 (5)	−0.0195 (5)	−0.0069 (5)
N1	0.0265 (6)	0.0217 (6)	0.0269 (6)	0.0018 (5)	−0.0089 (5)	−0.0073 (5)
N2	0.0305 (7)	0.0212 (6)	0.0303 (6)	0.0023 (5)	−0.0131 (5)	−0.0074 (5)
C1	0.0277 (8)	0.0324 (8)	0.0412 (9)	0.0015 (6)	−0.0071 (7)	−0.0214 (7)
C2	0.0479 (11)	0.0329 (8)	0.0393 (9)	0.0032 (8)	−0.0218 (8)	−0.0128 (7)
C3	0.0457 (10)	0.0248 (7)	0.0360 (9)	−0.0020 (7)	−0.0170 (7)	−0.0077 (7)
C4	0.0287 (8)	0.0234 (7)	0.0306 (8)	0.0018 (6)	−0.0084 (6)	−0.0117 (6)
C5	0.0353 (9)	0.0253 (7)	0.0313 (8)	0.0008 (6)	−0.0093 (7)	−0.0106 (6)
C6	0.0363 (9)	0.0252 (7)	0.0376 (8)	−0.0041 (6)	−0.0050 (7)	−0.0126 (7)
C7	0.0373 (10)	0.0442 (10)	0.0525 (11)	−0.0026 (8)	−0.0119 (8)	−0.0291 (9)
C8	0.0282 (8)	0.0235 (7)	0.0297 (7)	0.0019 (6)	−0.0097 (6)	−0.0088 (6)
C9	0.0236 (7)	0.0245 (7)	0.0264 (7)	0.0004 (6)	−0.0054 (6)	−0.0086 (6)
C10	0.0251 (7)	0.0229 (7)	0.0252 (7)	0.0010 (6)	−0.0052 (6)	−0.0075 (6)
C11	0.0324 (8)	0.0256 (7)	0.0294 (8)	0.0010 (6)	−0.0114 (6)	−0.0100 (6)
C12	0.0396 (9)	0.0226 (7)	0.0328 (8)	−0.0003 (6)	−0.0126 (7)	−0.0098 (6)
C13	0.0308 (8)	0.0235 (7)	0.0251 (7)	0.0025 (6)	−0.0069 (6)	−0.0055 (6)
C14	0.0333 (8)	0.0275 (7)	0.0316 (8)	0.0016 (6)	−0.0142 (7)	−0.0107 (6)
C15	0.0318 (8)	0.0236 (7)	0.0329 (8)	−0.0010 (6)	−0.0095 (6)	−0.0103 (6)
C16	0.0415 (10)	0.0290 (8)	0.0323 (8)	0.0039 (7)	−0.0175 (7)	−0.0095 (7)
C17	0.0419 (10)	0.0281 (8)	0.0329 (8)	0.0019 (7)	−0.0176 (7)	−0.0079 (7)
C18	0.0639 (13)	0.0373 (10)	0.0352 (9)	0.0030 (9)	−0.0061 (9)	−0.0090 (8)
C19	0.0788 (16)	0.0381 (10)	0.0358 (10)	−0.0077 (10)	−0.0143 (10)	−0.0021 (8)
C20	0.0710 (15)	0.0297 (9)	0.0589 (13)	0.0028 (9)	−0.0322 (11)	−0.0033 (9)
C21	0.0626 (15)	0.0408 (11)	0.0793 (16)	0.0178 (10)	−0.0088 (13)	−0.0120 (11)
C22	0.0490 (12)	0.0379 (10)	0.0505 (11)	0.0070 (9)	−0.0047 (9)	−0.0055 (9)
C23	0.111 (2)	0.0359 (12)	0.097 (2)	0.0088 (13)	−0.0446 (19)	0.0047 (13)

Geometric parameters (Å, º)

Ni1—O1^{i}	1.8363 (10)	C10—C15	1.389 (2)
Ni1—O1	1.8363 (10)	C10—C11	1.403 (2)
Ni1—N1	1.8677 (12)	C11—C12	1.378 (2)

Ni1—N1^{i}	1.8678 (12)	C11—H11	0.9500
O1—C9	1.3009 (18)	C12—C13	1.390 (2)
O2—C13	1.3736 (17)	C12—H12	0.9500
O2—C16	1.4383 (18)	C13—C14	1.392 (2)
N1—C8	1.2977 (19)	C14—C15	1.389 (2)
N1—N2	1.4030 (16)	C14—H14	0.9500
N2—C9	1.3145 (18)	C15—H15	0.9500
C1—C2	1.389 (2)	C16—C17	1.506 (2)
C1—C6	1.391 (2)	C16—H16A	0.9900
C1—C7	1.504 (2)	C16—H16B	0.9900
C2—C3	1.382 (2)	C17—C18	1.376 (3)
C2—H2	0.9500	C17—C22	1.378 (3)
C3—C4	1.402 (2)	C18—C19	1.396 (3)
C3—H3	0.9500	C18—H18	0.9500
C4—C5	1.393 (2)	C19—C20	1.360 (3)
C4—C8	1.460 (2)	C19—H19	0.9500
C5—C6	1.385 (2)	C20—C21	1.393 (3)
C5—H5	0.9500	C20—C23	1.518 (3)
C6—H6	0.9500	C21—C22	1.386 (3)
C7—H7A	0.9800	C21—H21	0.9500
C7—H7B	0.9800	C22—H22	0.9500
C7—H7C	0.9800	C23—H23A	0.9800
C8—H8	0.9500	C23—H23B	0.9800
C9—C10	1.479 (2)	C23—H23C	0.9800

O1^{i}—Ni1—O1	180.0	C12—C11—C10	120.54 (14)
O1^{i}—Ni1—N1	96.53 (5)	C12—C11—H11	119.7
O1—Ni1—N1	83.47 (5)	C10—C11—H11	119.7
O1^{i}—Ni1—N1^{i}	83.47 (5)	C11—C12—C13	120.22 (14)
O1—Ni1—N1^{i}	96.53 (5)	C11—C12—H12	119.9
N1—Ni1—N1^{i}	180.00 (5)	C13—C12—H12	119.9
C9—O1—Ni1	111.02 (9)	O2—C13—C12	114.99 (13)
C13—O2—C16	117.77 (12)	O2—C13—C14	124.86 (14)
C8—N1—N2	119.42 (12)	C12—C13—C14	120.15 (14)
C8—N1—Ni1	125.97 (11)	C15—C14—C13	119.15 (14)
N2—N1—Ni1	114.52 (9)	C15—C14—H14	120.4
C9—N2—N1	107.12 (12)	C13—C14—H14	120.4
C2—C1—C6	117.53 (15)	C14—C15—C10	121.40 (14)
C2—C1—C7	121.02 (16)	C14—C15—H15	119.3
C6—C1—C7	121.45 (15)	C10—C15—H15	119.3
C3—C2—C1	122.41 (16)	O2—C16—C17	107.78 (13)
C3—C2—H2	118.8	O2—C16—H16A	110.2
C1—C2—H2	118.8	C17—C16—H16A	110.2
C2—C3—C4	119.88 (15)	O2—C16—H16B	110.2
C2—C3—H3	120.1	C17—C16—H16B	110.2
C4—C3—H3	120.1	H16A—C16—H16B	108.5
C5—C4—C3	117.81 (14)	C18—C17—C22	118.55 (17)
C5—C4—C8	116.21 (13)	C18—C17—C16	120.82 (17)

C3—C4—C8	125.97 (14)	C22—C17—C16	120.61 (16)
C6—C5—C4	121.58 (15)	C17—C18—C19	120.5 (2)
C6—C5—H5	119.2	C17—C18—H18	119.7
C4—C5—H5	119.2	C19—C18—H18	119.7
C5—C6—C1	120.75 (15)	C20—C19—C18	121.3 (2)
C5—C6—H6	119.6	C20—C19—H19	119.3
C1—C6—H6	119.6	C18—C19—H19	119.3
C1—C7—H7A	109.5	C19—C20—C21	118.14 (18)
C1—C7—H7B	109.5	C19—C20—C23	121.1 (2)
H7A—C7—H7B	109.5	C21—C20—C23	120.7 (2)
C1—C7—H7C	109.5	C22—C21—C20	120.8 (2)
H7A—C7—H7C	109.5	C22—C21—H21	119.6
H7B—C7—H7C	109.5	C20—C21—H21	119.6
N1—C8—C4	130.93 (14)	C17—C22—C21	120.6 (2)
N1—C8—H8	114.5	C17—C22—H22	119.7
C4—C8—H8	114.5	C21—C22—H22	119.7
O1—C9—N2	123.84 (13)	C20—C23—H23A	109.5
O1—C9—C10	117.23 (12)	C20—C23—H23B	109.5
N2—C9—C10	118.93 (13)	H23A—C23—H23B	109.5
C15—C10—C11	118.51 (14)	C20—C23—H23C	109.5
C15—C10—C9	122.35 (13)	H23A—C23—H23C	109.5
C11—C10—C9	119.15 (13)	H23B—C23—H23C	109.5

N1—Ni1—O1—C9	−1.22 (10)	O1—C9—C10—C11	1.6 (2)
N1^{i}—Ni1—O1—C9	178.78 (10)	N2—C9—C10—C11	−179.24 (15)
O1^{i}—Ni1—N1—C8	5.45 (14)	C15—C10—C11—C12	−0.3 (2)
O1—Ni1—N1—C8	−174.55 (14)	C9—C10—C11—C12	179.90 (14)
O1^{i}—Ni1—N1—N2	−178.16 (10)	C10—C11—C12—C13	−1.1 (3)
O1—Ni1—N1—N2	1.84 (10)	C16—O2—C13—C12	172.90 (15)
C8—N1—N2—C9	174.67 (14)	C16—O2—C13—C14	−6.6 (2)
Ni1—N1—N2—C9	−1.98 (15)	C11—C12—C13—O2	−178.27 (15)
C6—C1—C2—C3	−1.5 (3)	C11—C12—C13—C14	1.3 (3)
C7—C1—C2—C3	178.71 (17)	O2—C13—C14—C15	179.45 (15)
C1—C2—C3—C4	2.2 (3)	C12—C13—C14—C15	0.0 (3)
C2—C3—C4—C5	−0.9 (3)	C13—C14—C15—C10	−1.4 (3)
C2—C3—C4—C8	178.31 (16)	C11—C10—C15—C14	1.5 (2)
C3—C4—C5—C6	−1.0 (3)	C9—C10—C15—C14	−178.65 (15)
C8—C4—C5—C6	179.76 (15)	C13—O2—C16—C17	−177.34 (14)
C4—C5—C6—C1	1.6 (3)	O2—C16—C17—C18	103.66 (19)
C2—C1—C6—C5	−0.4 (3)	O2—C16—C17—C22	−77.6 (2)
C7—C1—C6—C5	179.40 (16)	C22—C17—C18—C19	−0.6 (3)
N2—N1—C8—C4	0.3 (3)	C16—C17—C18—C19	178.15 (17)
Ni1—N1—C8—C4	176.52 (13)	C17—C18—C19—C20	0.6 (3)
C5—C4—C8—N1	−164.03 (17)	C18—C19—C20—C21	0.1 (3)
C3—C4—C8—N1	16.8 (3)	C18—C19—C20—C23	−178.8 (2)
Ni1—O1—C9—N2	0.40 (19)	C19—C20—C21—C22	−0.9 (4)
Ni1—O1—C9—C10	179.52 (10)	C23—C20—C21—C22	178.1 (2)
N1—N2—C9—O1	1.0 (2)	C18—C17—C22—C21	−0.2 (3)

N1—N2—C9—C10	−178.07 (12)	C16—C17—C22—C21	−178.91 (19)
O1—C9—C10—C15	−178.23 (14)	C20—C21—C22—C17	0.9 (4)
N2—C9—C10—C15	0.9 (2)		

Symmetry code: (i) −*x*+2, −*y*, −*z*+2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O1^{i}	0.95	2.38	2.9455 (18)	118
C3—H3···N2	0.95	2.37	2.945 (2)	118
C11—H11···O1	0.95	2.43	2.7590 (19)	100

Symmetry code: (i) −*x*+2, −*y*, −*z*+2.