Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[2-(dimethylamino)ethanol-κ^2N,O](pentane-2,4-dionato-κ^2O,O')nickel(II) chloride

Manzar Sohail,[a] Kieran C. Molloy,[b] Muhammad Mazhar,[a]* G. Kociok-Köhn[b] and M. Kaleem Khosa[a]

[a]Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan, and [b]Department of Chemistry, University of Bath, Bath BA2 7AY, England

Correspondence e-mail: mazhar42pk@yahoo.com

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.005 Å
R factor = 0.050
wR factor = 0.137
Data-to-parameter ratio = 19.9

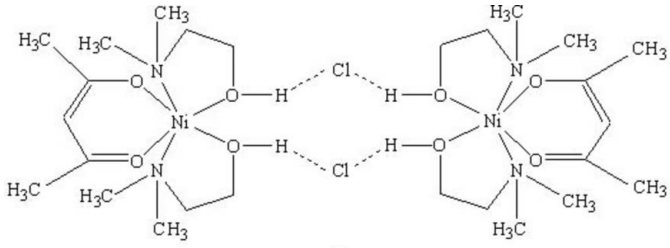
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The Ni atom in the title complex, $[Ni(C_5H_7O_2)(C_4H_{11}NO)_2]Cl$, is in a distorted octahedral coordination environment. Cations are linked into centrosymmetric dimers *via* O—H···Cl hydrogen bonds involving the OH groups of the 2-(dimethylamino)ethanol ligands and the Cl^- anions.

Received 30 June 2005
Accepted 9 September 2005
Online 17 September 2005

Comment

The title compound, (I), is a synthetic precursor for the possible deposition of nickel oxide thin films through aerosol-assisted chemical vapour deposition (AACVD). The molecular structure of complex (I) is shown in Fig. 1, and selected bond lengths and angles are given in Table 1.



(I)

The complex has a distorted octahedral geometry around the Ni^{II} atom and contains two bidentate chelating dimethylaminoethanol groups and a bidentate acetylacetonate group. The N atoms are in mutually *trans* positions, with an N2—Ni1—N1 angle of 171.43 (10)°. The Ni1—N2 bond length of 2.139 (3) Å is significantly shorter than that of 2.166 (3) Å for Ni1—N1. The Ni—O1, Ni—O2 and Ni—O3 bonds are very similar to the analogous bonds in the related compound $[Ni(acac)_2(dmaeH)]$ (acac is acetylacetonate and dmaeH is dimethylaminoethanol; Williams *et al.*, 2001). Not surprisingly,

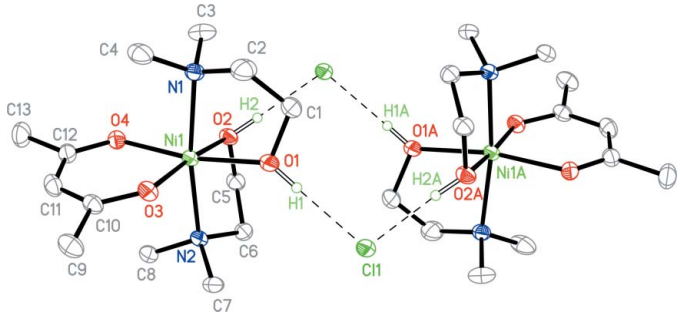


Figure 1
The hydrogen-bonded (dashed lines) dimer of the title compound, showing 30% displacement ellipsoids. Atoms labelled with the suffix A are related by the symmetry operator $(2-x, 1-y, 1-z)$.

the Ni—O bonds of the coordinated dmaeH groups are longer [2.080 (2) and 2.106 (2) Å] than the Ni—O(acac) bonds [2.014 (2) and 2.015 (2) Å]. The *cis* O—Ni—O and O—Ni—N bond angles in (I) are close to the ideal octahedral value of 90°, lying in the range 89.07 (9)–93.84 (9)°, with the exception of the bite angles of the chelating dmaeH groups [80.30 (10) and 81.10 (9)°], and 97.08 (10)° for N2—Ni1—O4. Distortions of the *trans* O—Ni—O angles from the ideal 180° are also evident [169.95 (9)–172.31 (10)°].

In the crystal structure, molecules are linked *via* O—H···Cl hydrogen bonds to form centrosymmetric dimers involving the O—H groups of the dmaeH ligands and the Cl^- anions [H1—Cl 2.15 (4) and H2—Cl1^{i} 2.18 (4) Å, and O1—H1···Cl1 172 (4) and O2—H2···Cl1^{i} 161 (6)°; symmetry code: (i) $2 - x$, $1 - y$, $1 - z$].

Experimental

Bis(2,4-pentanedionato)nickel(II), [Ni(acac)$_2$] (0.5 g, 1.95 mmol), was reacted with dimethylaminoethanol (dmaeH; 0.391 ml, 3.9 mmol) in the presence of methoxytin(II) chloride (0.7 g, 3.9 mmol), [ClSnOCH$_3$] in toluene under argon. The resulting product was recrystallized from tetrahydrofuran at 263 K to give crystals of [Ni(acac)(dmaeH)$_2$]Cl, (I).

Crystal data

[Ni(C$_5$H$_7$O$_2$)(C$_4$H$_{11}$NO)$_2$]Cl
M_r = 371.54
Monoclinic, $P2_1/a$
a = 13.6400 (3) Å
b = 8.7900 (3) Å
c = 15.2310 (5) Å
β = 100.6970 (10)°
V = 1794.40 (9) Å^3
Z = 4
D_x = 1.375 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25977 reflections
θ = 2.9–27.5°
μ = 1.25 mm^{-1}
T = 150 (2) K
Block, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.706, T_{max} = 0.886
27270 measured reflections
4059 independent reflections
3191 reflections with $I > 2\sigma(I)$
R_{int} = 0.093
θ_{max} = 27.5°
$h = -17 \rightarrow 17$
$k = -11 \rightarrow 11$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.137
S = 1.07
4059 reflections
204 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0683P)^2 + 1.8862P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.00 e Å^{-3}
$\Delta\rho_{min}$ = −0.86 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	2.080 (2)	Ni1—N1	2.166 (3)
Ni1—O2	2.106 (2)	Ni1—N2	2.139 (3)
Ni1—O3	2.014 (2)	O1—H1	0.86 (4)
Ni1—O4	2.015 (2)	O2—H2	0.86 (2)
N1—Ni1—N2	171.43 (10)	N2—Ni1—O2	81.10 (9)
O1—Ni1—O4	169.95 (9)	N2—Ni1—O3	91.48 (10)
O2—Ni1—O3	172.31 (9)	N2—Ni1—O4	97.08 (10)
N1—Ni1—O1	80.30 (10)	O1—Ni1—O2	90.96 (10)
N1—Ni1—O2	93.78 (9)	O1—Ni1—O3	91.42 (10)
N1—Ni1—O3	93.84 (9)	O2—Ni1—O4	89.07 (9)
N1—Ni1—O4	89.66 (10)	O3—Ni1—O4	89.86 (9)
N2—Ni1—O1	92.86 (10)		

H atoms on O atoms were located in a difference map and refined isotropically. C-bound H atoms were positioned geometrically and refined as riding, with C—H = 0.98–0.99 Å and with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for methyl H atoms. The highest peak is located 0.96 Å from atom C2 and 1.61 Å from atom N1.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *HKL SCALEPACK* (Otwinski & Minor, 1997); data reduction: *DENZO* (Otwinski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank the Pakistan Science Foundation, Islamabad 45320, Pakistan, for funding [contract/grant No. PSF/R&D/C-QU/Chem(218)].

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Nonius (1997). *KappaCCD Server Software*. Windows 3.11 Version. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen Germany.
Sheldrick, G. M. (2001). *SHELXTL/PC*. Version 6.12 Windows NT Version. Bruker AXS Inc., Madison, USA.
Williams, P. A., Jones C. A., Bickley, F. J., Steiner, A., Davies, O. H., Leedham. J. T., Impey. A. S., Garcia. J., Allen, S., Rougier A. & Blyr, A. (2001). *J. Mater. Chem.* **11**, 2329–2935.

supporting information

Acta Cryst. (2005). E**61**, m2001–m2002 [doi:10.1107/S1600536805028424]

Bis[2-(dimethylamino)ethanol-κ^2N,O](pentane-2,4-dionato-κ^2O,O')nickel(II) chloride

Manzar Sohail, Kieran C. Molloy, Muhammad Mazhar, G. Kociok-Köhn and M. Kaleem Khosa

S1. Comment

The title compound, (I), is a synthetic precursor for the possible deposition of nickel oxide thin films through aerosol-assisted chemical vapour deposition (AACVD). The molecular structure of complex (I) is shown in Fig. 1, and selected bond lengths and angles are given in Table 1.

The complex has a distorted octahedral geometry around the Ni^{II} atom and contains two bidentate chelating dimethylaminoethanol groups *and* a bidentate acetylacetonate group. The N atoms are in mutually *trans* positions, with an N2—Ni1—N1 angle of 171.43 (10)°. The Ni1—N2 bond length of 2.139 (3) Å is significantly shorter than that of 2.166 (3) Å for Ni1—N1. The Ni—O1, Ni—O2 and Ni—O3 bonds are very similar to the analogous bonds in the related compound [$Ni(acac)_2$(dmaeH)] (acac is acetylacetonate and dmaeH is dimethylaminoethanol; Williams *et al.*, 2001). Not surprisingly, the Ni—O bonds of the coordinated dmaeH groups are longer [2.080 (2) and 2.106 (2) Å] than the Ni—O(acac) bonds [2.014 (2) and 2.015 (2) Å]. The *cis* O—Ni—O and O—Ni—N bond angles in (I) are close to the ideal octahedral value of 90°, lying in the range 89.07 (9)–93.84 (9)°, with the exception of the bite angles of the chelating dmaeH groups [80.30 (10) and 81.10 (9)°], and 97.08 (10)° for N2—Ni1—O4. Distortions of the *trans* O—Ni—O angles from the ideal 180° are also evident [169.95 (9)–172.31 (10)°].

In the crystal structure, molecules are linked *via* O—H···Cl hydrogen bonds to form centrosymmetric dimers invoving the O—H groups of the dmaeH ligands and the Cl^- anions [H1—Cl 2.15 (4) and H2—$Cl1^i$ 2.18 (4) Å, and O1—H1···Cl1 172 (4) and O2—H2···$Cl1^i$ 161 (6)°; symmetry code: (i) $2-x, 1-y, 1-z$].

S2. Experimental

Bis(2,4-pentanedionato)nickel(II), [$Ni(acac)_2$] (0.5 g, 1.95 mmol), was reacted with dimethylaminoethanol (dmaeH; 0.391 ml, 3.9 mmol) in the presence of methoxytin(II) chloride, [$ClSnOCH_3$] in toluene under argon. The resulting product was recrystallized from tetrahydrofuran at 263 K to give crystals of [$Ni(acac)(dmaeH)_2$]Cl, (I).

S3. Refinement

H atoms on O atoms were located in a difference map and refined isotropically. C-bound H atoms were positioned geometrically and refined as riding, with C—H = 0.98–0.99 Å and with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for methyl H atoms. **[Please check added text and correct as necessary.]**

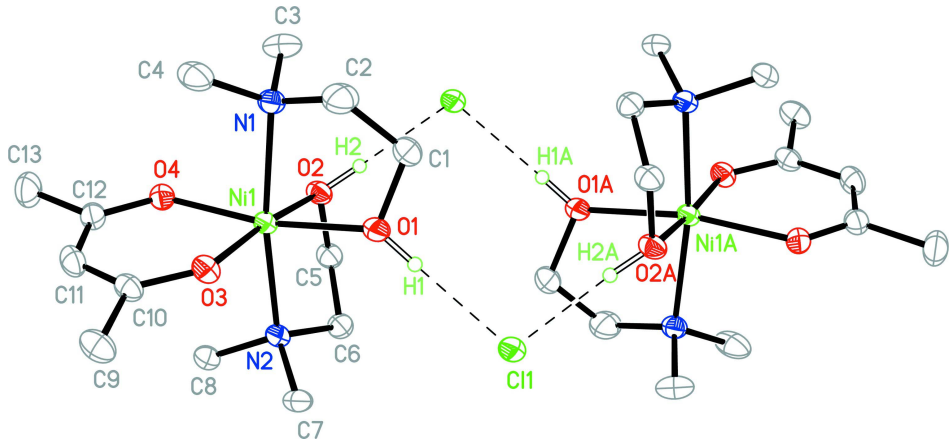


Figure 1

The hydrogen-bonded (dashed lines) dimer of the title compound, showing 30% displacement ellipsoids. The atoms labelled with the suffix A are related by the symmetry operator $(2 - x, 1 - y, 1 - z)$.

2,4-Pentanedionatobis(dimethylaminoethanol)nickel(II) chloride

Crystal data

$[Ni(C_5H_7O_2)(C_4H_{11}NO)_2]Cl$
$M_r = 371.54$
Monoclinic, $P2_1/a$
Hall symbol: -P 2yab
$a = 13.6400$ (3) Å
$b = 8.7900$ (3) Å
$c = 15.2310$ (5) Å
$\beta = 100.697$ (1)°
$V = 1794.40$ (9) Å^3
$Z = 4$
$F(000) = 792$
$D_x = 1.375$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25977 reflections
$\theta = 2.9$–27.5°
$\mu = 1.25$ mm^{-1}
$T = 150$ K
Plate, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker Nonius Kappa CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
188 2.0° images with ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{min} = 0.706$, $T_{max} = 0.886$
27270 measured reflections
4059 independent reflections
3191 reflections with $I > 2\sigma(I)$
$R_{int} = 0.093$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.6°$
$h = -17 \rightarrow 17$
$k = -11 \rightarrow 11$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.137$
$S = 1.07$
4059 reflections
204 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0683P)^2 + 1.8862P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.00$ e Å^{-3}
$\Delta\rho_{min} = -0.86$ e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.93608 (3)	0.33643 (4)	0.26988 (2)	0.02843 (14)
Cl1	0.86046 (6)	0.29196 (9)	0.56412 (5)	0.0374 (2)
O1	0.8803 (2)	0.4046 (3)	0.38181 (17)	0.0420 (6)
H1	0.881 (3)	0.371 (5)	0.435 (3)	0.048 (12)*
O2	1.07688 (16)	0.4291 (3)	0.32353 (15)	0.0344 (5)
H2	1.080 (5)	0.510 (4)	0.356 (3)	0.096 (19)*
O3	0.81020 (15)	0.2204 (3)	0.22212 (15)	0.0355 (5)
O4	0.98193 (16)	0.3064 (3)	0.15270 (14)	0.0340 (5)
N1	0.87266 (19)	0.5542 (3)	0.22294 (18)	0.0341 (6)
N2	1.00493 (19)	0.1378 (3)	0.33520 (17)	0.0319 (6)
C1	0.8531 (3)	0.5613 (4)	0.3789 (3)	0.0506 (9)
H1A	0.9128	0.6244	0.4008	0.061*
H1B	0.8039	0.5795	0.4181	0.061*
C2	0.8100 (3)	0.6035 (5)	0.2869 (3)	0.0577 (10)
H2A	0.8018	0.7154	0.2832	0.069*
H2B	0.7431	0.5568	0.2702	0.069*
C3	0.9500 (3)	0.6658 (4)	0.2141 (3)	0.0519 (10)
H3A	0.9185	0.7604	0.1890	0.078*
H3B	0.9926	0.6254	0.1744	0.078*
H3C	0.9905	0.6864	0.2731	0.078*
C4	0.8064 (3)	0.5424 (5)	0.1348 (3)	0.0600 (12)
H4A	0.7716	0.6393	0.1203	0.090*
H4B	0.7573	0.4614	0.1365	0.090*
H4C	0.8461	0.5185	0.0892	0.090*
C5	1.1457 (2)	0.3168 (4)	0.3682 (2)	0.0366 (7)
H5A	1.1936	0.3651	0.4170	0.044*
H5B	1.1838	0.2712	0.3254	0.044*
C6	1.0870 (2)	0.1952 (4)	0.4056 (2)	0.0365 (7)
H6A	1.1318	0.1101	0.4290	0.044*
H6B	1.0587	0.2376	0.4557	0.044*
C7	0.9365 (3)	0.0434 (4)	0.3773 (2)	0.0389 (7)
H7A	0.9737	−0.0409	0.4099	0.058*
H7B	0.8834	0.0026	0.3310	0.058*
H7C	0.9069	0.1060	0.4189	0.058*
C8	1.0469 (3)	0.0400 (4)	0.2719 (2)	0.0386 (7)

H8A	1.0961	0.0980	0.2463	0.058*
H8B	0.9931	0.0064	0.2240	0.058*
H8C	1.0792	−0.0489	0.3038	0.058*
C9	0.7069 (2)	0.0304 (4)	0.1439 (3)	0.0467 (9)
H9A	0.7143	−0.0350	0.1969	0.070*
H9B	0.7040	−0.0328	0.0905	0.070*
H9C	0.6452	0.0896	0.1387	0.070*
C10	0.7951 (2)	0.1375 (4)	0.1526 (2)	0.0350 (7)
C11	0.8532 (2)	0.1379 (4)	0.0866 (2)	0.0406 (8)
H11	0.8322	0.0742	0.0362	0.049*
C12	0.9396 (2)	0.2238 (4)	0.0880 (2)	0.0367 (7)
C13	0.9908 (3)	0.2209 (6)	0.0080 (3)	0.0557 (10)
H13A	0.9975	0.3250	−0.0131	0.084*
H13B	0.9509	0.1607	−0.0398	0.084*
H13C	1.0571	0.1751	0.0252	0.084*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0323 (2)	0.0229 (2)	0.0296 (2)	−0.00079 (14)	0.00439 (15)	−0.00080 (15)
Cl1	0.0490 (4)	0.0275 (4)	0.0339 (4)	−0.0053 (3)	0.0035 (3)	−0.0004 (3)
O1	0.0648 (16)	0.0258 (12)	0.0400 (13)	0.0061 (11)	0.0213 (12)	0.0017 (10)
O2	0.0356 (11)	0.0265 (12)	0.0377 (12)	−0.0014 (9)	−0.0019 (9)	−0.0019 (9)
O3	0.0350 (11)	0.0313 (12)	0.0405 (12)	−0.0028 (9)	0.0082 (9)	−0.0045 (10)
O4	0.0374 (11)	0.0330 (12)	0.0321 (11)	−0.0014 (9)	0.0079 (9)	−0.0015 (9)
N1	0.0378 (13)	0.0272 (13)	0.0355 (13)	0.0008 (10)	0.0020 (11)	0.0013 (11)
N2	0.0386 (13)	0.0235 (13)	0.0330 (13)	0.0004 (10)	0.0048 (11)	−0.0003 (10)
C1	0.073 (3)	0.037 (2)	0.045 (2)	0.0192 (18)	0.0180 (18)	−0.0015 (15)
C2	0.068 (3)	0.046 (2)	0.063 (3)	0.0188 (19)	0.022 (2)	0.0087 (19)
C3	0.049 (2)	0.037 (2)	0.068 (3)	−0.0023 (15)	0.0058 (19)	0.0187 (18)
C4	0.066 (2)	0.036 (2)	0.064 (3)	0.0086 (18)	−0.025 (2)	0.0002 (18)
C5	0.0344 (15)	0.0334 (18)	0.0391 (17)	0.0037 (12)	−0.0006 (13)	−0.0009 (13)
C6	0.0388 (16)	0.0342 (17)	0.0335 (16)	0.0039 (13)	−0.0014 (13)	0.0019 (13)
C7	0.0487 (18)	0.0263 (16)	0.0426 (18)	0.0000 (13)	0.0113 (14)	0.0062 (13)
C8	0.0483 (18)	0.0277 (16)	0.0391 (17)	0.0061 (13)	0.0066 (14)	−0.0008 (13)
C9	0.0351 (16)	0.0384 (19)	0.065 (2)	−0.0054 (14)	0.0042 (16)	−0.0076 (17)
C10	0.0301 (14)	0.0293 (16)	0.0429 (17)	−0.0003 (12)	−0.0001 (13)	−0.0038 (13)
C11	0.0398 (17)	0.039 (2)	0.0390 (17)	0.0001 (14)	−0.0025 (14)	−0.0102 (14)
C12	0.0420 (17)	0.0345 (17)	0.0326 (15)	0.0035 (13)	0.0042 (13)	−0.0022 (13)
C13	0.069 (3)	0.062 (3)	0.0397 (19)	−0.008 (2)	0.0213 (18)	−0.0101 (18)

Geometric parameters (Å, º)

Ni1—O1	2.080 (2)	C4—H4A	0.9800
Ni1—O2	2.106 (2)	C4—H4B	0.9800
Ni1—O3	2.014 (2)	C4—H4C	0.9800
Ni1—O4	2.015 (2)	C5—C6	1.510 (5)
Ni1—N1	2.166 (3)	C5—H5A	0.9900

Ni1—N2	2.139 (3)	C5—H5B	0.9900
O1—C1	1.425 (4)	C6—H6A	0.9900
O1—H1	0.86 (4)	C6—H6B	0.9900
O2—C5	1.442 (4)	C7—H7A	0.9800
O2—H2	0.86 (2)	C7—H7B	0.9800
O3—C10	1.270 (4)	C7—H7C	0.9800
O4—C12	1.273 (4)	C8—H8A	0.9800
N1—C3	1.465 (4)	C8—H8B	0.9800
N1—C2	1.475 (5)	C8—H8C	0.9800
N1—C4	1.476 (4)	C9—C10	1.515 (4)
N2—C7	1.481 (4)	C9—H9A	0.9800
N2—C8	1.483 (4)	C9—H9B	0.9800
N2—C6	1.487 (4)	C9—H9C	0.9800
C1—C2	1.463 (6)	C10—C11	1.391 (5)
C1—H1A	0.9900	C11—C12	1.397 (5)
C1—H1B	0.9900	C11—H11	0.9500
C2—H2A	0.9900	C12—C13	1.512 (5)
C2—H2B	0.9900	C13—H13A	0.9800
C3—H3A	0.9800	C13—H13B	0.9800
C3—H3B	0.9800	C13—H13C	0.9800
C3—H3C	0.9800		

N1—Ni1—N2	171.43 (10)	H3B—C3—H3C	109.5
O1—Ni1—O4	169.95 (9)	N1—C4—H4A	109.5
O2—Ni1—O3	172.31 (9)	N1—C4—H4B	109.5
N1—Ni1—O1	80.30 (10)	H4A—C4—H4B	109.5
N1—Ni1—O2	93.78 (9)	N1—C4—H4C	109.5
N1—Ni1—O3	93.84 (9)	H4A—C4—H4C	109.5
N1—Ni1—O4	89.66 (10)	H4B—C4—H4C	109.5
N2—Ni1—O1	92.86 (10)	O2—C5—C6	108.5 (3)
N2—Ni1—O2	81.10 (9)	O2—C5—H5A	110.0
N2—Ni1—O3	91.48 (10)	C6—C5—H5A	110.0
N2—Ni1—O4	97.08 (10)	O2—C5—H5B	110.0
O1—Ni1—O2	90.96 (10)	C6—C5—H5B	110.0
O1—Ni1—O3	91.42 (10)	H5A—C5—H5B	108.4
O2—Ni1—O4	89.07 (9)	N2—C6—C5	110.4 (3)
O3—Ni1—O4	89.86 (9)	N2—C6—H6A	109.6
C1—O1—Ni1	112.7 (2)	C5—C6—H6A	109.6
C1—O1—H1	109 (3)	N2—C6—H6B	109.6
Ni1—O1—H1	137 (3)	C5—C6—H6B	109.6
C5—O2—Ni1	112.66 (18)	H6A—C6—H6B	108.1
C5—O2—H2	109 (4)	N2—C7—H7A	109.5
Ni1—O2—H2	119 (4)	N2—C7—H7B	109.5
C10—O3—Ni1	126.0 (2)	H7A—C7—H7B	109.5
C12—O4—Ni1	126.1 (2)	N2—C7—H7C	109.5
C3—N1—C2	112.2 (3)	H7A—C7—H7C	109.5
C3—N1—C4	107.1 (3)	H7B—C7—H7C	109.5
C2—N1—C4	106.8 (3)	N2—C8—H8A	109.5

C3—N1—Ni1	111.8 (2)	N2—C8—H8B	109.5
C2—N1—Ni1	106.6 (2)	H8A—C8—H8B	109.5
C4—N1—Ni1	112.2 (2)	N2—C8—H8C	109.5
C7—N2—C8	107.9 (3)	H8A—C8—H8C	109.5
C7—N2—C6	109.2 (3)	H8B—C8—H8C	109.5
C8—N2—C6	109.6 (3)	C10—C9—H9A	109.5
C7—N2—Ni1	113.64 (19)	C10—C9—H9B	109.5
C8—N2—Ni1	111.10 (19)	H9A—C9—H9B	109.5
C6—N2—Ni1	105.37 (18)	C10—C9—H9C	109.5
O1—C1—C2	109.3 (3)	H9A—C9—H9C	109.5
O1—C1—H1A	109.8	H9B—C9—H9C	109.5
C2—C1—H1A	109.8	O3—C10—C11	125.1 (3)
O1—C1—H1B	109.8	O3—C10—C9	115.6 (3)
C2—C1—H1B	109.8	C11—C10—C9	119.2 (3)
H1A—C1—H1B	108.3	C10—C11—C12	125.6 (3)
C1—C2—N1	112.3 (3)	C10—C11—H11	117.2
C1—C2—H2A	109.1	C12—C11—H11	117.2
N1—C2—H2A	109.1	O4—C12—C11	125.5 (3)
C1—C2—H2B	109.1	O4—C12—C13	115.0 (3)
N1—C2—H2B	109.1	C11—C12—C13	119.6 (3)
H2A—C2—H2B	107.9	C12—C13—H13A	109.5
N1—C3—H3A	109.5	C12—C13—H13B	109.5
N1—C3—H3B	109.5	H13A—C13—H13B	109.5
H3A—C3—H3B	109.5	C12—C13—H13C	109.5
N1—C3—H3C	109.5	H13A—C13—H13C	109.5
H3A—C3—H3C	109.5	H13B—C13—H13C	109.5